RHEOLOGICAL STUDY OF SULPHUR MODIFIED BITUMINOUS BINDER

A THESIS SUBMITTED IN PARTIAL FULFILMENT OF THE REQUIRMENTS FOR THE DEGREE OF

MASTER OF TECHNOLOGY

In

CIVIL ENGINEERING

By

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CERTIFICATE

This is to certify that the thesis entitled, **“RHEOLOGICAL STUDY OF SULPHUR MODIFIED BITUMEN”** submitted by **Jhunarani Ojha** in partial fulfillment of the requirements for the award of Master of Technology Degree in Civil Engineering with Specialization in “Transportation Engineering” at National Institute of Technology, Rourkela, is an authentic work carried out by her under my supervision and guidance. To the best of my knowledge, the matter embodied in this Project Report has not been submitted to any other University/Institute for the award of any Degree or Diploma.

Date:

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DATE: **JHUNARANI OJHA**

ABSTRACT

The characteristic performance of asphalt concrete pavement always depends on the properties of bitumen, volumetric properties of asphalt concrete mixtures and external factors such as environment and traffic volume. Bitumen is a visco-elastic material where the temperature and rate of load application have a great influence on its behaviour. Conventional bitumen binder is exposed to a range of loading and atmospheric weather conditions; it is brittle in cold weather and soft in a hot environment. Within pavement layer higher traffic volume produces high stress, which is one of the main causes for pavement distress. The most serious distresses associated with flexible pavements are Fatigue cracking and permanent deformation. These distresses reduce the service life of the pavement and increase the cost of maintenance. There are different solutions to reduce the pavement distresses such as using asphalt additives or by adopting new mix design. Asphalt additive is known to give the conventional bitumen better engineering properties as well as it is helpful to extend the life span of asphalt concrete pavement.

In this work an investigation has been to use commercial sulphur available in local market to modify the conventional bitumen and attempt has been made to study the rheological properties of the modified binders prepared with different sulphur contents and at different mixing temperatures. This helps to understand the influence of modifiers on the rheological properties and fatigue resistance with the aim of preventing fatigue cracking in bituminous pavement. The conventional bitumen VG 30 viscosity grade was used in this work, modified with sulphur at five different modification levels namely 2%, 3%, 4%, 6% and 8% by weight of the bitumen. The rheological properties for asphalt binder were performed using a dynamic shear rheometer apparatus.

It is observed that the addition of 2% sulphur with VG 30 bitumen, prepared at 100^{o} C temperature results in the optimum rheological properties. Also the effects of aging on the rheological properties of bitumen and modified binder have been studied. It has been observed that the modified binders considered in this investigation satisfy the rutting and fatigue cracking.

***Keywords* -** viscosity grade bitumen, sulphur, rheology, dynamic shear rheometer, complex shear modulus, phase angle, amplitude sweep, frequency sweep.

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LIST OF ABBREVIATIONS

PAV: Pressure Ageing Vessel

TFO: Thin Film Oven

SHRP: Strategic Highway Research Program

DSR: Dynamic Shear Rheometer

AASHTO: American Association of State Highway and Transportation Officials

G' : Storage modulus [Pa] ,

G" : Loss Modulus [Pa]

G* : complex modulus

τ_{max} : Absolute value of the peak-to-peak shear stress(Pa)

γ_{max} : Absolute value of the peak-to-peak shear strain (%)

T_{max} : Maximum applied torque (load) (Pa)

θ_{max} : Maximum deflection angle (rad)

r : Radius of specimen plate (mm)

h : Specimen height (mm)

CHAPTER-1

INTRODUCTION

1.1 BACKGROUND

The term “bitumen” originated in Sanskrit, where the words “jatu” meaning pitch and jatu-krit meaning pitch creating referred to the pitch produced by some resinous trees. The Latin equivalent is claimed to be originally “gwitu-men” or “pixtu-men” which was shortened subsequently to “bitumen”.

Bitumen is one of human’s oldest engineering materials and is used at the beginning of civilization as a water proofing and bonding agent. In the middle of 19^{th} century to utilise rock asphalt for road surfacing so many attempts were made and due to this there was a slow development of the use of natural products, followed by the coal tar and later of bitumen manufactured from crude oil. It is defined in the Wikipedia as a mixture of organic liquids that are black, sticky, highly viscous, entirely soluble in carbon disulfide (CS_2), and composed primarily of aromatic hydrocarbons.

Although “natural” bitumen, such as Trinidad Lake Asphalt, are still used, most present day applications use the bitumen manufactured from crude oil. The manufacturing of bitumen from crude oil involves the processes of distillation, blowing and blending. The crude oil is a complex mixture of hydrocarbons, is refined by fractional distillation to separate gas, gasoline, kerosene, and long residue. The long residue is then further distilled to produce short residue, which is the used in the manufacturing of different grades of bitumen.

Although bitumen can be used for a wide variety of applications, the principal use is for the construction of highway and airport pavements, which together account for approximately 85% of the worldwide consumption of bitumen. The use of bitumen in combination with mineral aggregate to form an asphalt mixture results in a road construction material that not only has good qualities as a surfacing layer but, when correctly designed , provides the structural component layer of flexible pavement construction. The bitumen is not only an important engineering material but also a vital component in pavement engineering.

With the rapidly increasing Indian road transportation infrastructure, the road network is undergoing a challenging development under Bharat Nirman ,National Highways Development Programs (NHDP), Pradhan Mantri Gram Sadak Yojana (PMGSY), and State Highways Improvement Programs (SHIPs), etc. where a huge money is being invested by the Government of India in order to reach excellent pavement performance.

The flexible pavements, with top bituminous layers, are more preferred in India. These are economical with regard to both initial construction cost and maintenance costs. Bituminous binders commonly used in surface courses are unmodified binders such as VG-30and VG-10 bitumen (depending on the climatic conditions hot or cold) and modified binders such as CRMB. The present Indian specifications have clearly recommended the acceptance criteria for these binders by considering different physical tests which are all empirical in nature. The penetration test which is used to indicate hardness or softness of a binder. This test has no relation of bitumen to its actual field performance. There is great demand of investment on bituminous road infrastructures, so it is highly essential to think of use of performance based specifications of materials so that the ultimate objectives of investments should be fulfilled.

Asphalt concrete consists of asphalt (used as a binder) mixed with mineral aggregate and then laid down in layers and compacted. In order to achieve highway construction requirements designer should consider traffic flow, environmental factors and asphalt concrete mixtures materials. The design of pavements also requires two main material property considerations such as the load-deformation characteristics to calculate the stresses and strains in the pavement and the performance characteristics of material to determine mode of failures.

The mechanical properties of asphalt concrete mixtures are greatly affected by the bitumen binder properties; and the volumetric composition of aggregate, bitumen and air voids. Bitumen is a viscoelastic material. Visco-elastic means it behaves partly like an elastic solid (deformation due to loading is recoverable) and partly like a viscous liquid (deformation due to loading is not recoverable) and its behaviour depends on the temperature and rate of loading. it is known that conventional bitumen binder has a limited range of rheological properties which is not sufficient to resist pavement distresses. Therefore, material researchers looking for different types of modified bitumen with excellent rheological properties, which directly affect performance of asphalt pavement.

Recently, Strategic Highway Research Program (SHRP) introduced the performance based binder specification in total quality perspective based on the time and shear parameters, for all type of pavements failure. In India, the failure modes may be identified as critical pavement distresses resulting from fatigue cracking and rutting. Considering the above problems, an attempt is made in this project to study the performance related characteristics of one unmodified bitumen i.e. VG-30 bitumen and modified bitumen i.e. sulphur modified bitumen. The main objective of this study is to study the rheological characteristics of these binders. It is expected that this can explore the scope of development of new specifications for paving binders in India.

1.2 INDIAN SCENARIO AND ASSOCIATED PROBLEMS

In India, the bitumen grading is provided on the basis of penetration test, which is conducted to know the hardness or softness of bitumen at a temperature of 25°C, and does not indicate behaviour (viscous/elastic) of bitumen at the test temperature.

A huge sum of money is invested in highway construction to reach excellent pavement performance. But the pavement shows distress due to change in climatic condition and high traffic loads, which are directly affect the durability and pavement performance. The most common problem associated with the performance of bituminous pavements is fatigue cracking during cold climate and rutting during hot summer. Therefore, pavement distress needs urgent solutions which is very necessary and does not accept any delay.

The typical road surface temperature on a hot summer day is 60°C to 70°C. At this temperature the bitumen becomes soft and starts to penetrate and shove under loaded truck tyres which lead to rutting and corrugations under the wheel tracks of the highway pavement. But when temperature falls or in the cold climate, bituminous pavements become too brittle and fatigue cracks occur when excessively loaded. Fatigue is the process of cumulative damage resulting from repeated traffic loading. There are several ways to minimize the distresses of asphalt concrete pavements, which could improve the service and extent the pavement life such as:

• By producing a new binder with improved physical, chemical and rheological properties.

• By improving the pavements and mix design.

• By improving the construction methods and maintenance techniques.

The most important solutions for pavement distress are to develop a new binder with the help of an additive. There are several methods available to develop modified bitumen by using different additives. Sulphur has also been used as a common method to extend bitumen properties and is found to have wide range of application and potential for use.

1.3 RESEARCH OBJECTIVES

1. The aim of this work is to explore use of modified binder to improve the performance of flexible pavements.

2. The dynamic shear rheometer (DSR) is used to determine the rheological characteristics of bitumen binder over a wide range of temperature and rate of loding conditions.
3. Comparing the binder rheological properties at high, medium, low temperatures for unmodified bitumen as well as modified bitumen.
4. Study on effects of sulphur on modification of bitumen in terms of flow, amplitude sweep, frequency sweep, temperature sweep, creep response, creep recovery.
5. The changes of rheological properties have been studied before and after ageing using Rolling Thin Film Oven (RTFO) And Pressure Aging Vessel (PAV).

1.4 SCOPE OF THE WORK

The scope of the work emphasises on the rheological characteristics of unmodified (VG-30) and sulphur modified bitumen binder. The rheological tests and creep tests are conducted using a dynamic shear rheometer (DSR) apparatus based on the fundamental of dynamic mechanical analysis. Ageing of bitumen and modified binders has been understand using the Rolling Thin Film Oven (RTFO) for short term ageing and Pressure Aging Vessel (PAV) for long term ageing and effect of ageing on the rheological parameters are studied.

1.5 THESIS ORGANISATION

The thesis is presented in various chapters.

Chapter 1: presents an introductory chapter including the problem statement, the objectives of the research work; scope of the study and the layout of the thesis.

Chapter 2: provides a literature review starting with an introduction and followed by brief summary with regarding to composition and physical and chemical properties of bitumen.

Chapter 3: deals with theoretical considerations involved including bitumen rheology. This chapter reviews the fundamentals of dynamic shear rheometer and explains two important rheological parameters i.e. Complex shear modulus (G*) and phase angle (δ).

Chapter 4: contains experimental works dealing with short term ageing with the help of Rolling Thin Film Oven (RTFO) and long term ageing by Pressure Vessel Ageing (PAV). This chapter also contains methods to characterize the rheological behaviour of neat bitumen and the modified one.

Chapter 5: describes the rheology and fatigue laboratory test results for modified and unmodified binder (both aged and unaged).

Chapter 6: includes conclusions on the project work along with future scope of work followed by a list of references considered and reviewed.

CHAPTER-2

LITERATURE REVIEW

2.1 INTRODUCTION

The purpose of this chapter is to review the literature of rheological properties of bituminous pavements. This review consists of four parts; the first parts deals with the constitution of bitumen with regards to its elementary analysis and chemical composition.

The second parts deals with the measurement of bitumen rheology, from the conventional physical tests. The literature review gives a brief description of the conventional tests, such as softening point, penetration, specific gravity, ductility test and also a detailed description of the rheological characteristics and visco-elastic behaviour of bitumen. Also describes the various means to analyse the rheological data.

The third parts of the literature review describes about the common flexible pavement distresses. This is most important.

The fourth part deals with the modification of bitumen. The review describes the various types of additives and different modification mechanism.

The fifth part describes the aging of bitumen and its effect on rheology.

2.2 CONSTITUTION OF BITUMEN (CHEMICAL COMPOSITION)

2.2.1 Elemental Composition of Bitumen

The bitumen is defined as dark brown to black, sticky and viscous material which is composed of high molecular weight hydrocarbons. It is obtained from the bottom of the vacuum distillation columns in the crude oil refineries.

Whiteoak. (1990) describes the elementary analysis of bitumen manufactured from the various crude oils with varying physical properties. It predominately consists of carbon and hydrogen. But it shows that most bitumen binder contains heteroatom such as carbon, hydrogen, sulphur, oxygen and nitrogen. The elementary analysis of the bitumen binder is presented below in Table 2.1

Table 2.1 Elementary Analysis of Bitumen

COMPONENT	**PERCENTAGE %**
Carbon	82% -88%
Hydrogen	8% -11%
Sulphur	0% -6%
Oxygen	0% -1.5%
Nitrogen	0% -1%

2.2.2 Chemical Groups of Bituminous Materials

Robert et al (2000) mentioned that bitumen is consists of two major chemical groups called asphaltenes and maltenes. The major chemical composition for bitumen is presented below in 2.1:

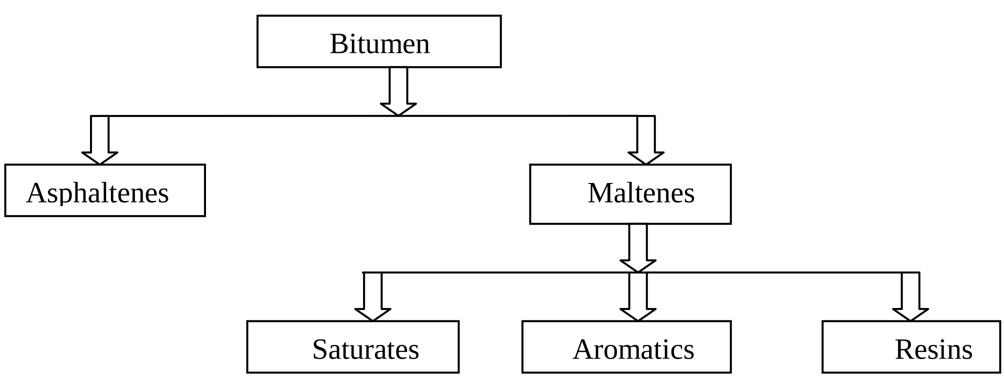

Figure 2.1 Chemical Groups of Bitumen [Robert et al (2000)]

Asphaltenes: Asphaltenes are dark brown friable solids with highest polarity and insoluble in non-polar solvents. Bitumen with higher asphaltenes content will have more viscosity and lower penetration.

Maltenes: As described before maltenes is divided into three groups, such as saturates, aromatics and resins.

Saturates: Saturates are straight and branched chain hydrocarbons. These are non-polar viscous oils, white in colour, with similar molecular weight range to aromatics.

Resin: Resins are defined as dark semi solid material having lower molecular weight. When heated it is liquid and brittle at low temperature. It acts as dispersing agents to the asphaltenes.

Aromatics: These are the lowest molecular weight compounds in bitumen. These are dark brown viscous liquids.

2.3 BITUMEN RHEOLOGY

2.3.1 Definition of Rheology

Rheology is the study of flow and deformation of material under applied mechanical forces**.** To measure the physical properties of bitumen, the primary importance is given to the characterisation of the rheologcal behaviour of bitumen binder. The stiffness of bitumen is time dependant, it means the flow with time. It also temperature dependent.so both time and temperature of loading to be considered when characterise the flow properties of rheoogical materials as bitumen. Bituminous material deforms when subjected to loads and the properties of bituminous material change with change of temperatures during day and night. It has been well established that the rheological properties of the bitumen binder affect the asphalt pavement performance.

Vinogradov et al (1980) describes the rheology as a part of continuum mechanics and the study of material flow and deformation. Rheology is the description of the mechanical properties for different materials under various deformation conditions.

Bahia and Davies (1994) mentioned and used the rheological properties as an indicator of flexible pavement performance. The rheological properties are related to the permanent deformation i.e. rutting at high temperature. The rheological properties at intermediate temperature are related to the fatigue cracking. Improved fatigue life, Reduced rutting, and lower the low-temperature stiffness values have been measured in asphalt mixtures made with binders with improved rheological properties.

Anderson et al (1994) explained that the rheological properties of asphalt binder express an important role in asphalt concrete pavement performance. There are many asphalt pavement distresses, which are related to the rheological properties of asphalt binder. The fundamental rheological characterisation of the modified and unmodified asphalt binder can be used to predict asphalt pavement performance. These rheological properties of asphalt binder can be evaluated with the help of dynamic shear rheometer (DSR) apparatus. Different tests can be used to characterize the viscous and elastic behaviour of asphalt binder at high and intermediate service temperatures.

Bahia et al (1993) conducted a time sweep test using dynamic shear rheometer. The test provides a simple method of applying repeated cycling of stress or strain loading at selected temperatures and loading frequency. The initial data collected were very promising and showed that the time sweeps are effective in measuring binder damage behaviour under repeated loading in shear. The advantage of time sweep test that can be used to calculate fatigue life of asphalt binder based in dissipated energy approaches.

Bahia and Anderson (1995) describe the purpose and scope of the dynamic shear rheometer test. The dynamic shear rheometer (DSR) used to characterize the viscoelastic behaviour of bituminous material at intermediate and high service temperatures. Stress-strain behaviour defines the response of materials to load. Asphalt binder's exhibit aspects of both elastic and viscous behaviours; hence they are called viscoelastic materials.

Airey (1997) used dynamic shear rheometer apparatus to evaluate the polymer modified and aged bituminous binders properties. The result of these tests can be used to evaluate the rheological changes related with aging of SBS polymer modified binder. This result in an increased viscous behaviour of after aging compared to elastic behaviour of unmodified bitumen.

En. Nur et al (2011) defined rheology is the study and evaluation of the flow and permanent deformation time temperature dependant materials. The rheological properties are presented in terms of complex modulus (stiffness) and phase angle (viscoelastic) master curves. The rheological data of bitumen is presented in the following forms:

- Isochronal plot:-A curve on a graph representing the behaviour of the system at a constant frequency.

  Ex.-curves of complex modulus as a function of temperature at constant frequency
- Isothermal plot:- a curve representing the behaviour of a system at constant temperature.

  Ex: - curve of complex modulus as a function of frequency at constant temperature.
- Black diagram: - a graph of complex modulus versus phase angles.
- Cole-Cole diagram: - a graph of loss modulus as a function of storage modulus

- Master curves: - the relationship between temperatures and frequencies represented in curve known as maser curve.

2.3.2 Conventional Physical Properties Test

The bitumen is available in a variety types and grades. To judge the suitability of these binders various physical tests conducted such as penetration test, ductility test, softening point test, specific gravity test, and viscosity test.

Penetration test:

The penetration test determines the hardness or softness of bitumen by measuring the depth in tenths of a millimetre to which a standard loaded needle will penetrate in five seconds under a specified load at a fixed temperature of 25° C. It measures the consistency of the bitumen. Therefore lower the penetration value harder the bitumen and vice versa. This test can be considered as an indirect method of measurement of the viscosity of the bitumen.

ASTM D5 / D5M-13 describes the standard test method for penetration of bituminous materials.

Ductility test:

The ductility test measures the adhesive property of bitumen and its ability to stretch. The ductility is expressed as the distance in centimetres to which the standard briquette of bitumen can be stretched before the thread breaks. The test is conducted at a fixed temperature 27° C and a fixed rate of pull of 50mm per minute.

ASTM D113 - 07 describes the standard test method for ductility of bituminous materials

Softening Point Test:

The softening point is the temperature at which the substance attains a particular degree of softening under specified condition of test. Generally higher softening point indicates the lower temperature susceptibility and preferred in warm climates.

ASTM D36 / D36M - 12 describes the standard test method for softening point of bitumen (ring-and-ball apparatus).

Specific Gravity Test:

The specific gravity of bituminous materials is defined as the ratio of the mass of a given volume of the substance to the same of an equal volume of water, at 27° C temperatures. It gives the density of bitumen. The density of bitumen is greatly influenced by its chemical composition. Increased amounts of aromatic compounds cause increase in specific gravity.

ASTM D70-03 describes the standard test method for specific gravity and density of semi-solid bituminous materials (pycnometer method).

The above conventional tests of defining the rheological properties of bitumen binder cannot be completely describe the viscoelastic properties. So these tests are not adequate, there is needed for the complete rheological evaluation of bitumen binder.

2.3.3 Viscoeastic Behaviour Of Bitumen

The viscoelastic behaviour refers to the mechanical properties of bituminous binder, which, in between two limiting extremes, the bitumen behaving likes as an elastic solid or as an viscous liquid depending on time of loading and temperature. Viscoelastic material is defined as the material, which stores and dissipates mechanical energy in response by a mechanical stress.

Robert (2000) explained that the Bitumen is a viscoelastic material and its behaviour depends on the rate of loading and temperature. At low temperature and small loading time bitumen behave as elastic solids. At high temperature and long rate of loading the bitumen behave as liquid. But at intermediate temperature and rate of loading the bitumen behaviour is viscoelastic, which is more complex and typical condition. The response of elastic, viscous and viscoelastic material under constant rate of loading are presented in figure 2.2. Figure 2.2(a) shows a constant stress is applied to an elastic material, the resulting strain of the material is proportional to the applied stress and when the applied load is withdrawn, the deforming strain is disappear i.e. there is a complete regain to its original position. Figure 2.2(b) shows the behaviour of a viscous material in which the deformation of the material increases over time under constant load. Figure 2.2(c) explains the behaviour of a viscoelastic material in which a constant stress increases the strain over a long time and when the applied stress is removed, the material fails to attain its original position, which leads to permanent deformation.

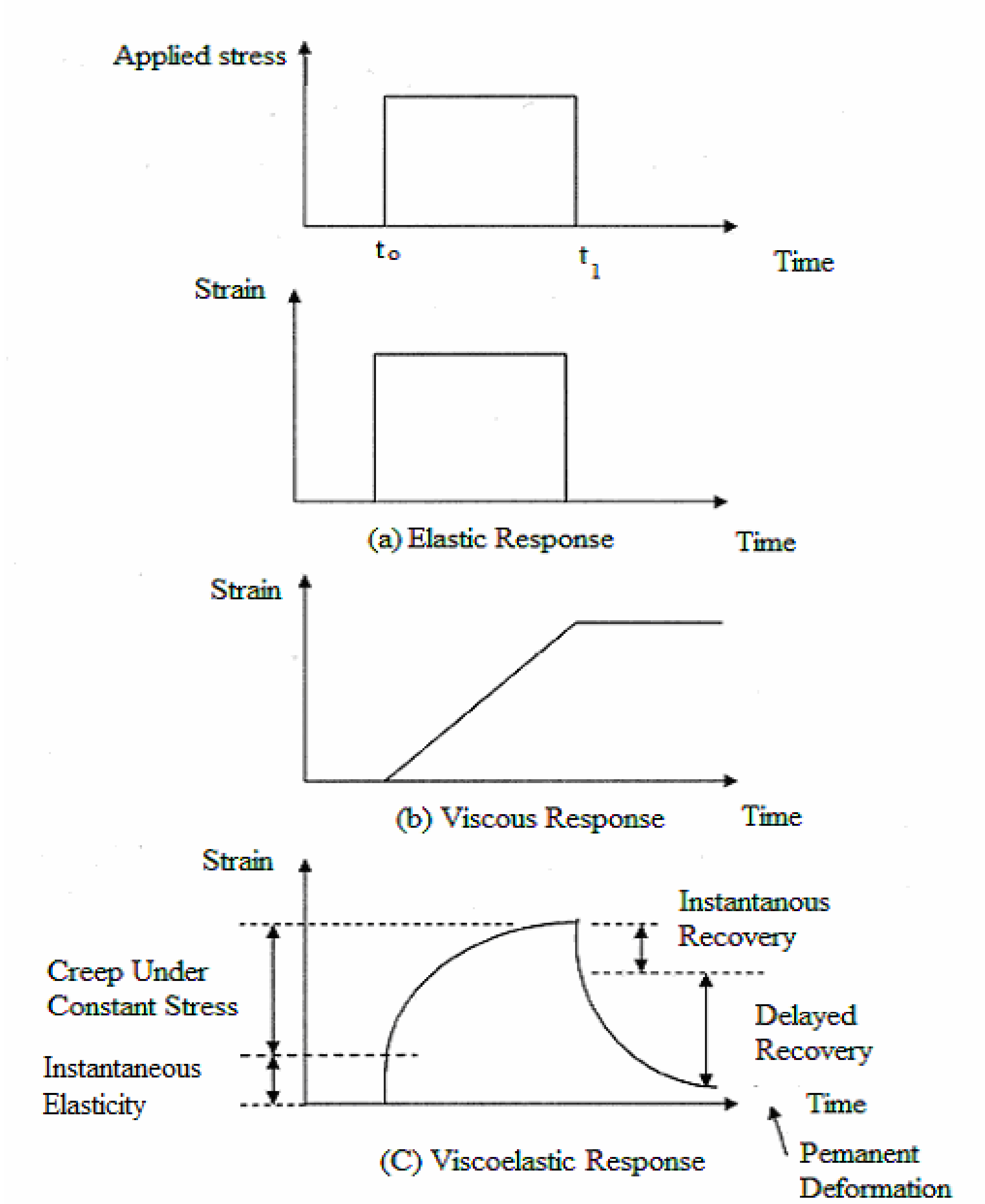

Figure 2.2 Idealized responses of elastic, viscous and viscoelastic material under constant stress loading (Van der Poel, 1954).

2.4 BITUMEN AGING

Aging of bitumen is a very complex process, which leads to hardening of bitumen resulting in a decrease in penetration and increase in softening point. In aging process the bitumen is affected by the oxygen, ultraviolet radiation and changes in temperature.

Anderson (1994) describes the hardening of the binder occurs in two different stages. The first stage is due to loss of binder volatile components during mixing which is called short-term aging, and is considered as the highest aging stage. The second stage is due to the oxidative hardening during service life which is called long term aging and related to the source or the chemical composition of the original binders.

There are four important mechanism of bitumen hardening:

- Loss of volatiles,
- Oxidation,
- Steric Hardening, and
- Exudative hardening (loss of lighter bitumen fractions by absorption into the aggregate)

Following laboratory test methods have been designed to both short term aging long term aging.

Rolling Thin Film Oven Test:

The rolling thin film oven (RTFO) test is used to stimulate the short term aging of asphalt binders that occurs, during the hot mixing process. In the test 35gm of bitumen taken in the bottle and placed inside the RTFO with airflow on and carriage roatating for 85 minutes at 163^{o} C temperatures. The residue from RTFO then tested for penetration, softening point and rheological properties.

ASTM D2872 - 12 describes the standard test method for effect of heat and air on a moving film of asphalt (rolli0ng thin-film oven test).

Pressure Aging Vessel:

The SHRP t0eam developed the method to use the pressure aging vessel (PAV) to simulate the physical an0d chemical property changes which in bitumen called long term aging or in-service oxidative 0aging after 5 to 10 years in field. The method involves oxidation of the bitumen in the RTFOT 0followed by the oxidation of the residue in a pressure aging vessel. The PAV test consists0 of aging 50 gm of bitumen placed in a pan within a pressurized heated close vessel with air to 2.1 MPa for 20 hours at 100^{o} C temperatures.

2.5 ASPHALT PAVEMENT DISTRESSES

Terrel (1971) explained that to improve the fatigue cracking performance of asphalt pavements it is important to understand the cracking mechanism of asphalt pavements. Fatigue failure is occur due to flexural cracking of asphalt bound layer. There are various factors affect the fatigue mechanism such temperature, rate of loading and aging. The complex interaction of these variables leads to use advanced mechanics theories such as damage mechanics, viscoelasticity, and fracture mechanics to understand the failure

Monismith et al (1985) divided the structural damage of flexible pavements in two main forms: cracking and deformation, which are produced due to load repetitions or adverse climatic conditions. Fatigue cracking decreases the service life of pavement and lead to pavement structure collapse. Different factors affect pavement performance and lead to pavement distress such as magnitude and frequency of loads density, duration of load cycle and variation of temperatures.

Lytton et al (1994) mentioned that fatigue cracking is a two stage process the first one is crack initiation and the second is crack propagation. Crack initiation is defined as a process by which cracks initiate on the asphalt pavement. Crack propagation is defined as the period for the cracks to grow and develop to form a critical size leading to pavement distress. The pavement distress and failure is considered as complex as several factors associate to the pavement deterioration and failure. The main causes of the pavement failure are:

- Defects in the quality of materials used.
- Defects in the construction method and quality control during construction.
- Surface and subsurface drainage.
- Settlement of the pavement foundation
- Environmental factors including heavy rainfall, snow, frost action and high water content

Miller and Bellinger (2003) mentioned asphalt pavement distresses are grouped into three types such as cracking, surface deformation, and surface defects. There are various reasons for asphalt concrete damage such as disintegration of mixture, fracture and visco plastic flow. The classifications of asphalt pavement distress are presented in Table 2.2

Table 2.2: Common Flexible Pavement Distresses (Miller and Bellinger, 2003)

Category	Distress type
Cracking	Longitudinal, Fatigue, Transverse, Reflective, Block, Edge
Deformation	Rutting, Corrugation, Shoving, Depression, Overlay Bumps
Surface defects	Potholes, Patching, Ravelling, Stripping

Bahia (2006) explained that fatigue cracking of flexible pavements is due to the horizontal tensile strain at the bottom of asphalt concrete layer. In the stage of crack initiation water trapped in the cracks and this led to reduction of the materials strength under repeated loading. Due to the strength reduction crack start to propagate and lead to pavement collapse.

Figure 2.3 Fatigue cracking of asphalt pavement (Bahia, 2006)

2.6 BITUMEN MODIFICATION

Conventional bitumen performs satisfactory applications in most highway pavement and airport pavement. However, in recent years, traffic levels, new axle designs, larger and heavier trucks, and tyre pressures severely increased on the highway system. When the bitumen does not meet the climatic condition, increased traffic and pavement structure requirements, at that time modification required for the enhancement of the properties of existing material. The main objective of the bitumen modification is to produce improved modified bitumen's materials with high resistance to fatigue cracking, and permanent deformation.

2.6.1 The Need of Asphalt Additives

There are so many researchers looking for the reasons to modify bituminous materials. **Lewandowski (1994)** mentioned that the important reasons to modify bituminous materials with various types of additives summarized as follows:

- To increase the stability and the strength of mixtures,
- To reduce rutting and, reach stiffer blends at high temperatures,
- To reduce cracking and, obtain softer blends at low service temperatures,
- To improve fatigue resistance of blends,
- To reduce thickness of flexible pavements.

King et al (1986) explained bitumen modifier as material, which would normally be added to the binder or the mixtures to improve its properties. For a particular project the choice of modifier can depend on so many factors including availability, construction ability, cost, and expected performance.

Robert et al (1991) described that the reasons for using modifiers in asphalt concrete mixtures are to resist rutting, produce stiffer mixes at high service temperature as well as to minimize thermal creaking, produce softer mixtures at low temperature and improve fatigue resistance of asphalt pavement. Improvement in the performance of asphalt concrete mixtures that contain polymer is largely due to the improvement in the rheological properties of the asphalt binder. The rheological properties of a binder that allow flexibility under load controls resistance to fatigue. The modified mixtures are less brittle at lower temperatures and it has higher stiffness at higher temperatures compared to normal mixtures. This makes polymer modification extremely attractive for pavement designers and highway agencies.

Brule (1996) described that Polymer modification increases binder stiffness and elasticity at high service temperatures and low loading frequencies with the degree of modification being a function of bitumen source, bitumen polymer compatibility and polymer concentration.

2.6.2Classification of Bitumen Additives

There are 3 type of bitumen modifier such as Physical modification, Chemical modification and Other type of modification

Table 2.3 Type of physical modifier and additive used in the material (Read and Whiteoak, 2003).

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	TYPE OF MODIFIER	TYPE OF ADDITIVE
1	Thermoplastic Elastomers	• Styrene-butadine-styrene(SBS) • Styrene-butadine-rubber (SBR) • Crumb tyre rubber • Polybutadine(PBD)
2	Thermoplastic polymer	• Ethylene vinyl acetate(EVA) • Ethylene methyl acrylate(EMA) • Polyethylene • Polypropylene • Polyvinyl chloride(PVC)
3	Thermosetting polymers	• Epoxy resin • Acrylic resin • Phenolic resin

Table 2.4 Type of chemical modifier and additive used in the material (Read and Whiteoak, 2003)

	TYPE OF MODIFIER	TYPE OF ADDITIVE
1	Chemical modifier	• Organo-metallic compounds • Sulphur • Lignin
2	Fiber	• Cellulose • Gass fiber • Asbestos • Polyester
3	Adhesive improvers	• Organic amines • Amides
4	Fillers	• Carbon black • Hydrated lime • Lime • Flyash
5	Extender	• Sulphur

2.6.3 Benefits of Using Asphalt Additives

Whiteoak (1990) describes are two methods to modify bitumen properties. The first method is to stiffen the bitumen so that the total visco-elastic response of the asphalt is reduced. The second option is to increase the elastic component of the bitumen, which reduces the viscous component of the bitumen and directly affect the pavement performance. Modified asphalt mixtures were observed to be stiffer, more resistant to permanent deformation, and had higher resistance to fatigue cracking. The area of asphalt additives is a somewhat complex, that the improvement in the pavement performance is related to the binder rheology and depend on the modifier type with respect to polymer content.

Bahia (1995) studied effect of polymer modification using scanning electron microscope images. The result showed that the modified asphalt concrete mixtures have better binder-aggregate adhesion, which led to increase in its toughness. Polymer modification affects the binder's flexibility that leads to fatigue resistance and increases the viscosity of the asphalt binder, which improve the tensile, and the compressive strengths of the mixtures. The role of modified bitumen is to increase the resistance of asphalt to permanent deformation at high temperatures.

2.7 SULPHUR AS ADDITIVE

Fritchy et al (1980) studied the bitumen modified with 2% of sulphur at 160^{o} C resulting more plastic consecutive to a variation of the nature of the interaction between asphaltenes molecules. The modification level consists of two phases such as phase I and phase II.

Phase –I: - In this phase various % of sulphur by weight is blended with neat bitumen at a particular temperature and rheological properties are defined. If the phase angle (δ) and complex shear modulus (G*) have significance difference than neat bitumen then the optimum % of sulphur modifier content was determined. Then keeping the optimum % of sulphur as constant the blending temperature should vary and rheological tests are done. From the rheological properties the optimum binder content with blending temperature is determined. Then proceed to phase –II.

Phase-II:- second phase start to evaluate the conventional bitumen properties, mechanical properties , rutting and fatigue resistance for modified and unmodified asphalt concrete mixtures.

Papirer et al (1980) conducted a study on the morphology of bitumen-sulphur mixes has been analysed by electron microscopy, after thin section.

Fatani and Sultan (1982) conducted a study to determine the feasibility of using dune sand in asphalt-concrete pavement in hot, desert like climates through the use of one size crushed aggregates. Dense graded aggregate and powdered sulphur were used in the sand asphalt mixes.

Arora and Rahman (1985) have explored the use of sulphur as a rejuvenation agent in recycling reclaimed asphalt pavement from a typical failed segment of Dammam-Abu=Hadriyah Expressway.

Akili (1985) carried out an extensive laboratory testing program designed to measure improvements in engineering properties of sulphur-asphalt-sand (SAS) mixes attributable to the presence of sulphur in the mix considering locally available sands and prevailing environmental conditions in eastern Saudi Arabia.

Mohamed (2007) conducted a study on the physical and mechanical properties of asphaltic concrete incorporating crumb rubber produced through dry process

Mohammed et al (2010) conducted a study on the sulphur extended asphalt as a major outlet for sulphur that outperformed other asphalt mixes in the Gulf.

Strickland et al conducted a Study on the Low-Temperature Properties of Sulphur Extended Asphalt Mixtures This study examines low temperature effects of SEA by using the Thermal Strength Restrained Specimen Test and also examines the effects of sulphur binder content as measured by stiffness modulus in both high and low temperatures. The study concludes that SEA can enhance the performance of HMA in both high and low temperature conditions.

The Federal Highway Administration (FHWA) completed a field study to compare the performance of sulphur-extended asphalt (SEA) pavements to conventional asphalt control (AC) pavements. The primary conclusion was that there was no difference in overall performance between the SEA and AC sections. Sulphur did not increase or decrease most test properties, and often it had no effect on a given test property of a mixture. Sulphur did decrease the resistance to moisture susceptibility in the laboratory. There were also minor trends indicating that with some mixtures, sulphur may reduce the susceptibility to rutting and increased the susceptibility to fatigue cracking

David and Mary (2009) conducted study on incorporation of sulphur extended asphalt mix in pavement design. It concludes that Thiopave has the potential to reduce the overall required pavement depth while still controlling strain at the bottom of the pavement

Bailey and Allen (2009) describes about the development innovative sulphur technology applied to asphalt mixtures. The concentration of the modified sulphur pellets in the mixture is designed to enhance asphalt mixture properties maintaining workability and compatibility. The performance of conventional asphalt base mixtures was compared with sulphur modified mixtures. The results concluded that the asphalt mixtures containing the modified sulphur pellets were showed improved performance compared to the conventional asphalt mixtures..

Richard (2010) carried a study on flexible pavement material stiffness significantly influences fatigue cracking and rutting performance. Therefore, choosing high-modulus asphalt concrete has the potential to increase the overall life of the pavement.

Colange et al (2010) shows that it is preferred to use an analytical method, which accounts for stiffness and fatigue rather than using a method that only utilises stiffness, as it can propose a layer thickness reduction, which can have a detrimental effect on pavement life.

Taylor et al (2010) conducted a study and describes hot liquid sulphur was control mix and 2nd generation sulphur-modified asphalt mixtures with varying binder replacement levels. Laboratory performance tests were conducted to measure the resistance of these mixes to moisture damage, permanent deformation, fatigue cracking, and low-temperature cracking. The results of the study showed that the sulphur-modification resulted in a tangible increase in stiffness over the control mixture when tested for dynamic modulus. This increase in stiffness afforded these mixes superior rut resistance versus the control mix in Asphalt .

CHAPTER -3

BITUMEN RHEOLOGY

3.1 INTRODUCTION

Bitumen is a viscoelastic liquid that behaves like elastic solid at low temperatures and rapid rate of loading (short loading times – high loading frequencies) and as a viscous fluid at high temperatures and slow rate of loading (long loading time -low frequencies). Therefore the response of bitumen to stress depends on both loading time and temperature. Bitumen rheology is defined as the fundamental measurements related with the flow and deformation of material under applied stress.

3.2 EVALUATION OF BITUMEN PROPERTIES

Bahia et al (1993) described that the research conducted for the Strategic Highway Research Program (SHRP), a new testing method introduced to characterize the rheological, failure, and durability properties of asphalt binders completely based on the rheological properties. The research results were discussed in four main points:(i) The viscoelastic nature of bitumen and its relation to performance of pavement; (ii) the fundamental problems related to these tests and; the types of conventional measurements are used now (iii) the concept of selecting the new test methods and the new characteristic properties; and (iv) how to compare the new measured properties to the conventional properties.

Rheological properties are used as performance parameter has advantages and disadvantage. The advantage is that it allows measurement of physical properties with wide temperature range at high and low frequency, which is likely to be experienced in the field due to traffic. Dynamic shear rheometer need qualified person with high experience to operate the dynamic tests and also to get good rheological results. In this chapter a brief description of the dynamic shear rheometer (DSR) apparatus as well as the geometry and specimen fabrication and specimen dimension will be presented. In this chapter also a detail description of all rheological test procedures adopted for the characterization of materials are given.

3.3 DYNAMIC SHEAR RHEOMETRY

Dynamic shear rheometer (DSR) was used to measure visco-elastic properties, fatigue cracking and rutting resistance at low, intermediate and high temperature. It measures both viscous and the elastic properties of the asphalt binders. DSR is also defined as a binder characterization procedure and used to determine the rheological properties of asphalt binders. This device used where sinusoidal shear stress or strain is applied in the form of sinusoidal time function to make dynamic oscillatory load. DSR device was used to measure various binder properties of unmodified and modified binder.

The principles involved in dynamic shear rheometer testing are shows in figure 3.1, where the bitumen sample is sandwiched between two parallel plates. The upper plate geometry is allowed to rotate about its own axis while base plate remains fixed during testing. The oscillation gives one smooth, continuous cycle which can be repeated continuously during the test. DSR tests are carried out over a wide range of frequencies (i.e. number of cycles per second) and temperature. The dynamic load can be given as sinusoidal time function, which is presented in the following equation

$$\tau = \tau_0 \,\mathrm{Sin}\,(\omega T) \qquad 3.1$$

Dynamic shear Rheometer is used to determine the specimen's response to the dynamic load. DSR tests can be carried out in two testing modes such as controlled stress or controlled strain. In the controlled strain mode, a specified magnitude of shear strain is applied to the bitumen and resultant shear stress is calculated. In the controlled stress mode, a specified magnitude of shear stress is applied to the bitumen and resultant shear strain is calculated.

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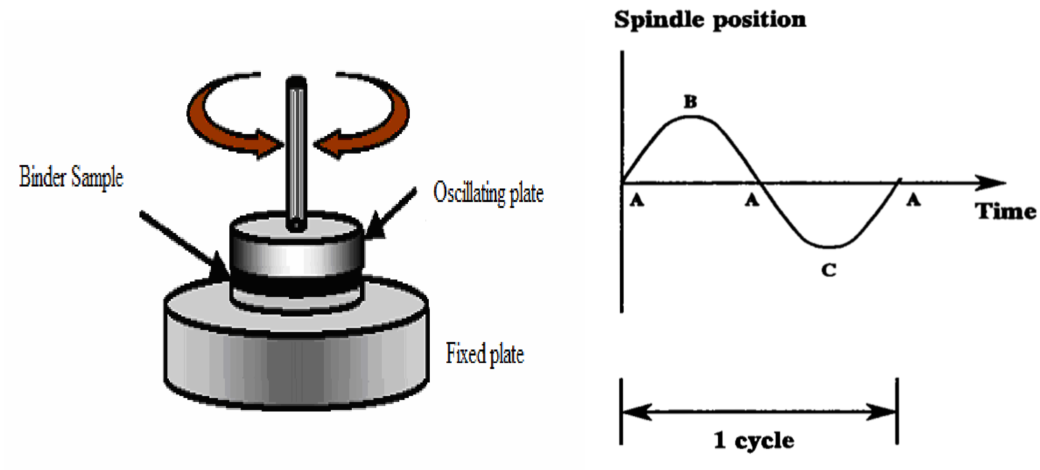

Figure 3.1 Principles of Operation of Oscillatory Type Dynamic Shear Rheometer

3.3.1 Theory of Analysis and Data Collection

The analysis of the tested sample can be presented as elastic or viscous component in the form of rheological parameter complex modulus and phase angle. The data acquisition unit records the test temperature, loading frequency, applied load and deflection angle during the test cycles, which directly sends the test data to the personal computer. The computer software calculates the rheological parameters such as shear stress shear strain, phase angle and complex modulus and present it in the form of table and figure.

Goodrich (1988) expresses the effect of frequency and temperatures on the viscosity and stiffness of the bitumen's tested sample. The test simulates the shearing action of traffic at a certain speed and determines two important parameters used to predict pavement performance. Rheological parameter are divided into two terms, the first one is the phase angle (δ) and the second one is the complex shear modulus (G*).

Gebhard, (2004) described that dynamic tests provide data on viscosity and elasticity related to the rate of applied load and temperature. DSR is a circular specimen mounted between two circular plates. The lower plate is constantly the fixed and upper plate rotate around a vertical axis. The specimens are subjected to specific shear stresses or strain with a range of frequencies by transfer the resulting torque to the upper plate. Dynamic testing gives an indication of the tested sample on resistance to deformation. The performance can be presented as elastic or viscous component in the form of two rheological parameter complex shear modulus (G*) and phase angle (δ).

3.3.2 Rheological Properties

Briscoe (1987) explained the benefit of the dynamic shear rheometer (DSR) that is used to characterize the elastic and viscous behaviour of bitumen binder at intermediate and high service temperatures. The purpose of the experiment was to study the effects of modifier type and optimum content on the rheological properties of bitumen binder.

Anderson et al (1994) described that the rheology of material is the study and evaluation of the time/temperature dependent response of materials, with an applied force. The rheological properties of the bitumen have a great influence on the properties between asphalt and aggregate. All DSR tests were done to evaluate the rheological properties with the environmental and rate of loading conditions of the pavement. The fundamental rheological parameters for modified and unmodified bitumen can be used as pavement performance indicator directly.

3.3.2.1 Phase Angle (δ)

Phase angle is defined as time lag between applied stress and resulting strain; it can be used to describe the viscoelastic behaviour of asphalt binder.

Bahia et al (1995) defined phase angle as the immediate elastic and the delayed viscous responses of the binder, obtained from the lag between the induced shear stresses and measured strains. In oscillatory deformation test the time lag between applied stress and resulting strain is shown graphically in figure (3.2). There is no phase difference between applied stress and resulting strain were found in elastic materials. At higher frequency and low temperature small phase angles are found since the bitumen approximates elastic behaviour. On the other hand, at lower frequency and high temperature higher phase angle are exhibited since the bitumen nearly viscous.

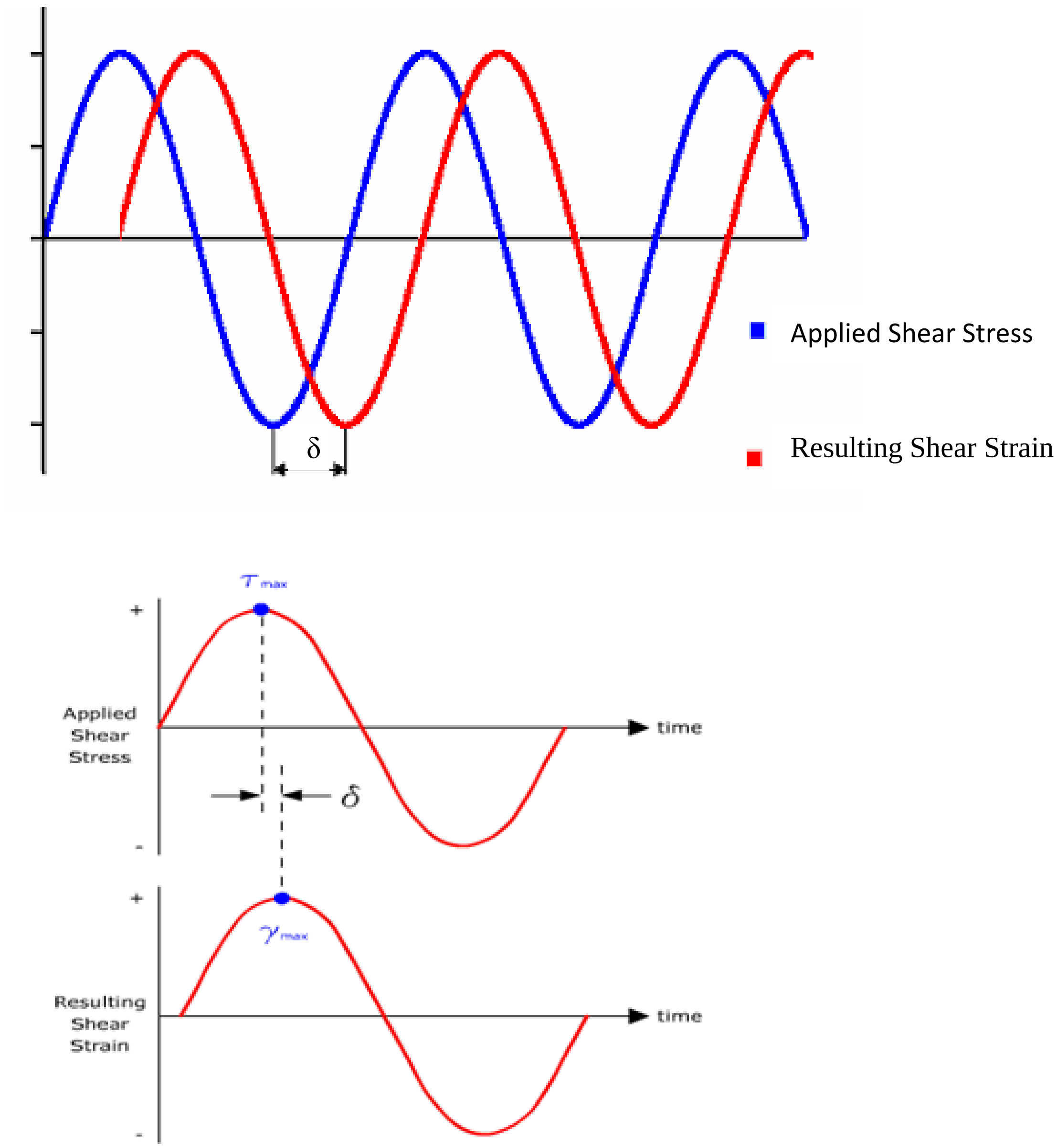

Figure 3.2 Viscoelastic material behaviour for dynamic sinusoidal loading

3.3.2.2 Dynamic Complex Shear Modulus (G*)

In controlled shear or controlled strain modes of rheological test, two main rheological parameter obtained i.e. the complex shear modulus (G*) and phase angle (δ). The complex shear modulus is defined as the total resistance to deformation when the bitumen binder is subjected to repeatedly shear loading. It is comprised of two components such as elastic component and viscous component, which are also known as the storage modulus (G′) and loss modulus (G″).

- Storage modulus (G′):- storage modulus can be defined as the elastic (recoverable) component. It is related to the amount of energy stores in the sample during each cycle of testing.
- Loss modulus (G″):- loss modulus can be defined as the viscous (non-recoverable) component, and also related to the energy lost during every testing cycle through permanent flow or deformation.

Bahia et al (1995) explained the relationship between the complex shear modulus (G*), storage modulus (G′), loss modulus, (G″) and phase angle (δ). If a substance is purely viscous then the phase angle (δ) is 90° that means storage modulus G’ = 0 and G’’ = G*. If a substance is purely elastic then the phase angle (δ) is zero that means G’ = (G*) and loss modulus G’’ = 0. There are numerous studies for using the rheological techniques to predict pavement performance based on the main two rheological parameter complex shear modulus and phase angle. The graphical relationship presented in figure (3-3) and described mathematically shown in following equation (3-2) to (3-6).

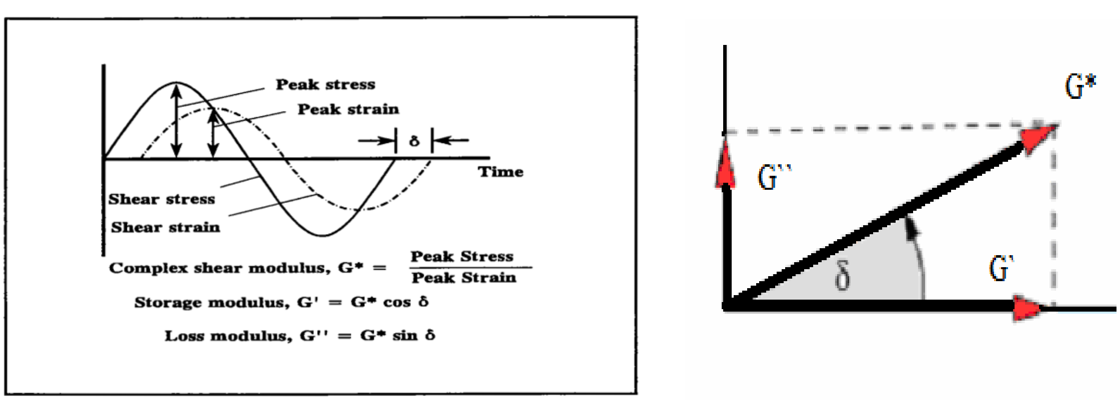

Figure 3.3 Relationship between Complex Shear Modulus (G*), Storage Modulus (G′), Loss Modulus (G″), and Phase angle (δ) (Bahia, 1993)

The axes of the figure 3.3 represent the bitumen behaviour. The vertical axis represents the completely viscous behaviour and the horizontal axis represents the completely elastic behaviour. At moderate to high temperature elastic behaviour can be achieved and at low temperature viscous behaviour realised. Under normal conditions of temperature and rate of loading found such a behaviour of bitumen which lies between the two axes and represented by a vector magnitude complex modulus (G*) and the direction of phase angle δ degrees anti clockwise from the horizontal axis.

$$G^* = (\tau_{max} - \tau_{min}) / (\gamma_{max} - \gamma_{min}) \quad \text{Pa} \qquad 3.2$$

$$G' = \text{Cos}\,(\delta)\,(\tau/\gamma) \qquad 3.3$$

$$G'' = \text{Sin}\,(\delta)\,(\tau/\gamma) \qquad 3.4$$

$$\tan(\delta) = G'/\,G'' \qquad 3.5$$

$$\tau_{max} = 2T_{max} / \Pi r^3 \qquad 3.6$$

$$\gamma_{max} = \theta_{max}\, r / h \qquad 3.7$$

Where:

G' : Storage modulus [Pa] ,

G" : Loss Modulus [Pa]

G* : complex modulus

τ_{max} : Absolute value of the peak-to-peak shear stress(Pa)

γ_{max} : Absolute value of the peak-to-peak shear strain (%)

T_{max} : Maximum applied torque (load) (Pa)

θ_{max} : Maximum deflection angle (rad)

r : Radius of specimen plate (mm), h : Specimen height (mm)

3.4 TEST SPECIMENS

The rheological tests were done according to the AASTHO: T 315-08(Determining the rheological Properties of Asphalt Binder Using a Dynamic Shear Rheometer (DSR)). This test method contains the determination of the dynamic shear modulus and phase angle of bitumen binder when tested in dynamic (oscillatory) shear using parallel plate test geometry. This test is applicable for the bitumen binders having dynamic shear modulus values in the range from 100 Pa to 10 MPa obtained between 6 to 88° C with an angular frequency of 10 rad/s. Dynamic shear rheometer, temperature unit and data acquisition unit are presented in figure (3.4)

Figure 3.4 Dynamic Shear Rheometer

3.5 BINDERS USED

Bitumen binders used in this study were (VG-30 and sulphur modified VG 30 bitumen) viscosity grade bitumen, which is the most widely used in India.

3.6 Specimen geometry

The specimen geometry was chosen according to the test condition, type and specification. The 25mm diameter specimen geometry with thickness (1mm) used for high temperature test to save the specimen from melting. At intermediate temperature the specimen should have small diameter (8mm) with sample thickness (2mm) to prevent it from brittle crack.

25 mm diameter - 1mm thickness
High temperature

8 mm diameter - 2mm thickness
Intermediate Temperature

Figure 3.5 (DSR) plates test samples for high temperature and intermediate temperatures

3.7 FACTORS AFFECTING DSR RHEOLOGICAL TESTING

3.7.1 Temperature

Bitumen binder is a very good thermal insulator and also highly temperature susceptible. To makes accurate measurements it is most important, firstly, the whole sample is at the same temperature and secondly, the temperature should accurately controlled. Fluid (water) bath temperature control system is used with dynamic shear rheometer.

3.7.2 Strain Amplitude, Stress Level and Frequency Of Oscillation

A viscoelastic material, bitumen does not behave linearly in terms of their stiffness as a function strain or stress. Therefore the phase angle and dynamic shear modulus depend upon the magnitude of the shear strain with both increasing and decreasing shear strain. A linear region is defined as at small strains where the complex shear modulus is independent of shear strain.

The limit of the linear viscoelastic behaviour is defined as the measured value of G* decreases to 95% of its zero strain value. The rheological tests should be performed with in the linear viscoelastic region of bitumen behaviour. So a strain sweep test was conducted at 60⁰ C as shown in figure 3.6 for VG-30 and sulphur modified bitumen.

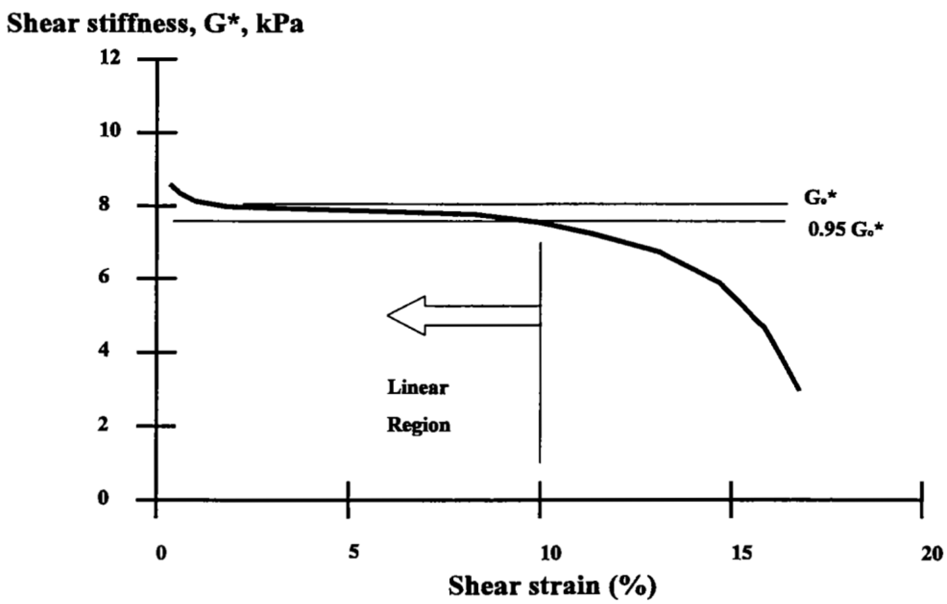

Figure 3.6 strain sweep used to determine linear viscoelastic region (Airey,)

3.7.3 Sample Preparation and Geometry

According to the AASTHO method the bitumen samples were prepared for the DSR testing. The sample geometry of the test sample consists of two parameters such as plate or disk diameter and gap width (thickness of specimen).

3.8 SUMMARY

Bahia (1995a) explained clear definition for oscillation and dynamic shear rheometer test summary were presented. Oscillation tests also known as dynamic tests which are used to calculate rheological parameter and to evaluate the response of specimen's to the sinusoidal stresses. Phase angle and Complex shear modulus of a binder are the indicators of asphalt's resistance to shear deformation, help to predict pavement distress such as rutting and fatigue. Viscoelastic properties of asphalt are determined by evaluating the behaviour of asphalt specimen, when it is subjected to oscillatory stress. Data acquisition unit records the test temperature, applied torque, loading frequency and deflection angle every 10 cycles of the test and send the data to the personal computer. The computer software calculates the shear stress, shear strain, complex modulus and phase angle. The software presents the measured and calculated value in the form of tables and figure

<u>CHAPTER-4</u>

EXPERIMENTATION

4.1 MATERIAL

It is known from the studies that the degree of modification depends on the neat bitumen type and modifier type. Various studies have been done in the field of sulphur modification and there are several explanations for the need of using modifier in bitumen binder industry. There are various reasons for using bitumen modifier in bitumen industry started with increase the service life of the pavement, improve the pavement performance, meet the heavy traffic demands, and finally saving the cost of maintenance. Physical and chemical properties of neat bitumen presented in Table 4.1.

Table 4.1 Physical properties of neat bitumen

PROPERTY	RANGE
Absolute viscosity 60°c poise min	2400
Flash point min. °c	220
Softening point min. °c	47
Penetration 0.1 min.25 °c	50-70
Ductility on min .25°c	40

The performance of asphalt concrete pavement can be improved by using sulphur modified bitumen. Bitumen binder used in this study was (VG-30) viscosity grade which is the most widely used for intermitted temperature. After the base bitumen was chosen, the second step for bitumen modification is looking for suitable modifier. In this study sulphur was used as to compare rheological properties of the produced binder with the mix design.

Table 4.2 Physical properties of sulphur modifier

Property	Range
specific gravity	1.957
Melting point	110^{o} C
boiling point of	444.674°C
Colour	Yellow

Figure 4.1 Elemental Sulphur

4.1.1 Preparation of Sulphur Modified Bitumen

The following blending sequence was used to modify bitumen materials with sulphur:

- Bitumen binder was heated in an oven at a temperature of at least 140 ^{o}C.
- The stainless steel cylindrical container capacity 1kg used for mixing was cleaned and kept in the oven at a temperature of at least 140 ^{o}C.
- The required amount 500 gm of asphalt was weighed into the container; then the amount of additive required to yield the desired additive-to-asphalt ratio was weighed.

- Five blends were prepared with 2%, 3%, 4%, 6%, and 8% sulphur respectively, by total weight of bitumen.
- The mixing temperature was controlled during mixing using heater and it is 100 ^{o}C for first set of sample preparation.
- The stirring was started by manually with the help of glass rod, and the prepared amount of additive was added gradually to the beaker while stirring.
- The stirring was continued for 10 min.
- The ready sulphur modified bitumen was used to prepare the tests sheet, which were used for making DSR specimen with different diameter using special tools.
- From the rheological properties the optimum % of modifier evaluated.
- Then above procedure were repeated for preparing sample with optimum % of modifier at various blending temperature of 95 ^{o}C, 105 ^{o}C, 115 ^{o}C, 125 ^{o}C, 135 ^{o}C respectively.

4.1.2 AGING OF BINDER

4.1.2.1 Short Term Aging

The rolling thin film oven (RTFO) test is used to stimulate the short term aging of asphalt binders that occurs, during the hot mixing process. In the test 35gm of bitumen taken in the bottle and placed inside the RTFO with airflow on and carriage rotating for 85 minutes at 163^{0} C temperatures. The residue from RTFO then tested for penetration, softening point and rheological properties.

Figure 4.2 Rolling Thin Film Oven for short term aging

4.1.2.2 Long Term Aging

The SHRP team developed the method to use the pressure aging vessel (PAV) to simulate the physical and chemical property changes which in bitumen called long term aging or in-service oxidative aging after 5 to 10 years in field. The method involves oxidation of the bitumen in the RTFOT followed by the oxidation of the residue in a pressure aging vessel. The PAV test consists of aging 50 gm of bitumen placed in a pan within a pressurized heated close vessel with air to 2.1 MPa for 20 hours at 100^0 C temperatures. After 20 hours of treatment of pressure agingthe samples are removed, degassed at 170° C and used for future testing.

Figure 4.3 Pressure Aging Vessel

Figure 4.4 Vacuum Oven For Degassing

4.2 TESTING PROGRAMME

4.2.1 Bitumen Binder Rheology Tests

The rheological properties, rutting and fatigue resistance tests were performed using the unmodified bitumen and modified bitumen. The various tests were performed under various ranges of stress, frequency and temperatures as follows:

1. Characterization of binders under standard conditions of SHRP

2. Amplitude Sweep Test (stress sweep)

3. Frequency Sweep Test at constant stress

4. Temperature Sweep Test

4.2.1.1 Characterization of Binders under Standard Conditions of SHRP

The test is conducted under certain condition which is given by SHRP used to characterize the bituminous binders. The test conditions are applied stress 120 Pa, and frequency 10 rad/sec performed at 60°C temperature, with the 25mm diameter plate having specimen thickness 1mm for unaged neat bitumen and RTFO aged binders. For PAV aged binders, the test temperature was changed to applied stress 50000 Pa, frequency 10 rad/sec performed at 40° C temperature with 8mm diameter plate having specimen thickness 2mm.

4.2.1.2 Amplitude Sweep Test (Stress Sweep)

A stress sweep was first conducted to determine the linear viscoelastic region for different binders. The main cause of the test within the linear viscoelastic region is that the relation between strain and stress is influenced only by the temperature or frequency and not by the magnitude of the stress or strain because the behaviour of the material is linear. The test was conducted at 60°C temperature for unaged and RTFO aged binders and 40 ° C temperature for PAV aged binders with a shear stress range from 1Pa to 15 kPa and at constant frequency of 10 Hz. The complex shear modulus G* versus stress/strain plot was used to determine the linear viscoelastic region (LVR). The stress determine from this test used as the parameter for further tests.

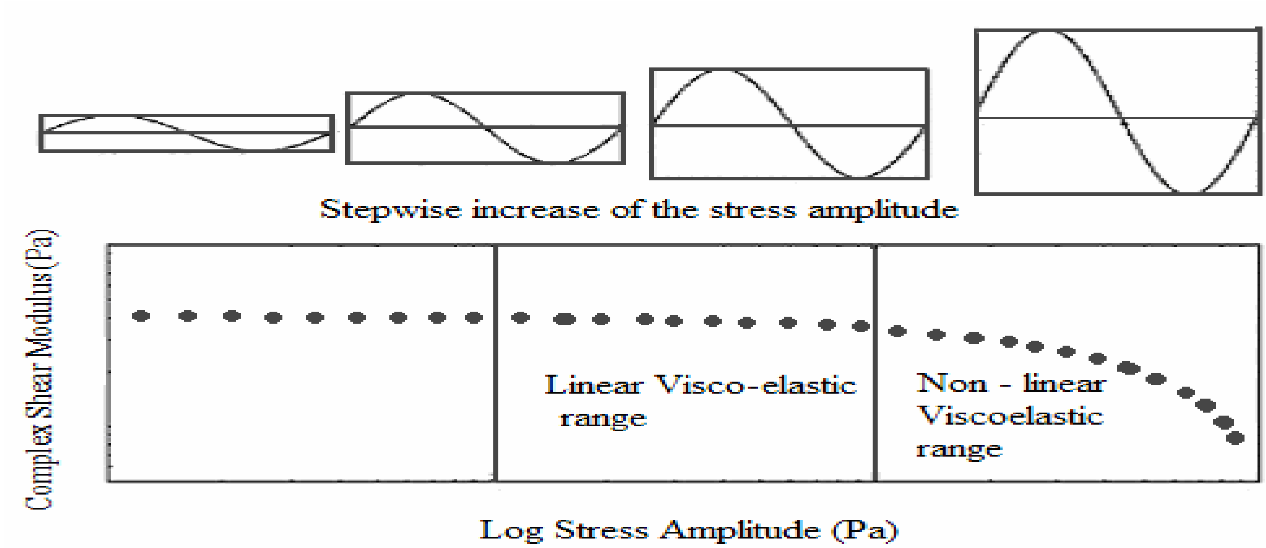

Figure 4.5 Amplitude stress sweep test after (Gebhard, 2004)

Gebhard, (2004) described that stress sweep is used to characterize the stress – strain response of the bituminous material. It is easy to describe the difference between elastic, viscous and viscoelastic behaviour according to the material response through this test.

4.2.1.3 Frequency Sweep Test

Frequency sweep tests were performed on unaged and modified bituminous binders. The test was conducted at 60°C temperature for unaged and RTFO aged binders and 40° C temperature for PAV aged binders. All of the frequency sweep tests were performed from 1 to 10 rad/s under a stress parameter taken from amplitude test. Frequency sweep test is used to construct the black diagrams and stiffness master curve for the tested sample.

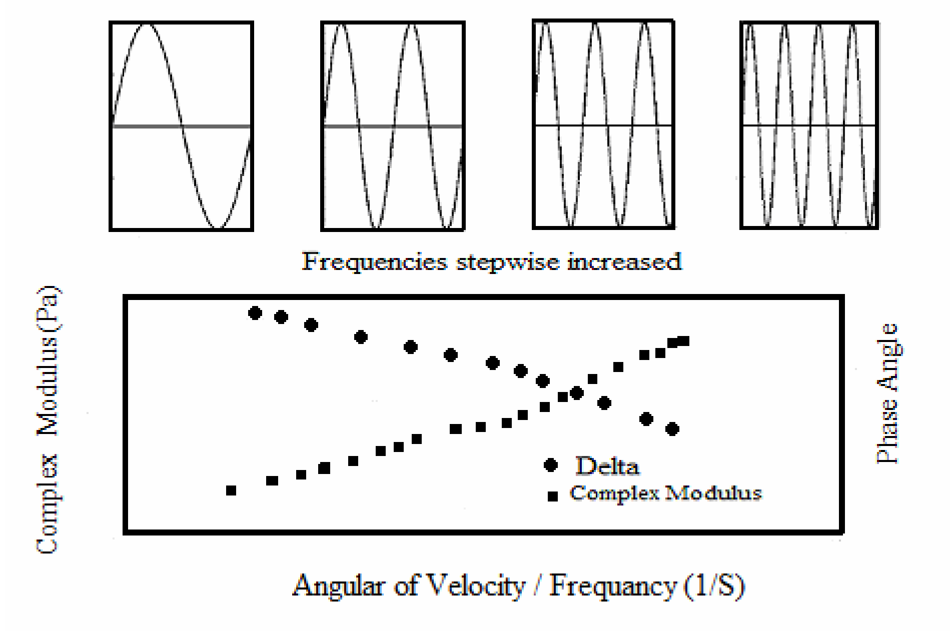

Figure 4.6 Frequency Sweep Test after (Gebhard, 2004)

4.2.1.4 Temperature Sweeps Test

This test was conducted by using a temperature variation starting from 25ºC to 65ºC, while the frequency was fixed at 10 Hz under a stress. Parallel plates with 8 mm diameter and 2 mm gap were used.

CHAPTER-5

ANALYSIS OF RESULTS

5.1 **INTRODUCTION**

This chapter describes the rheological properties, rutting, and fatigue behaviour results for unmodified bitumen and modified bitumen binder as well as brief analysis of test data. The framework of testing binders was selected in order to investigate the influence of sulphur in the bitumen properties subjected to different loading parameters. The rheological properties of the different binders were characterized using dynamic shear rheometer over wide ranges of temperatures and frequencies. In this study both neat bitumen and modified was tested and a summary of all results presented below in tables and graphical form.

5.2 THE RHEOLOGICAL CHARACTERISATION OF UNAGED BINDER

5.2.1 The Rheological Characterisation of Unaged Binder on Variation of % of Sulphur

5.2.1.1Characterization of Binders under Standard Conditions of SHRP

- The test was conducted under certain standard conditions given by SHRP which is used to characterize the bituminous binders.
- The following specifications are provided by SHRP for different binders:

➢ To control possible tenderness, the stiffness value $G^*/\sin(\delta)$ of the original binder must be greater than 1.0kPa at the average pavement temperature.

➢ To minimize rutting, the stiffness value $G^*/\sin(\delta)$ of the binder after RTFO must be greater than 2.2kPa at the average pavement design temperature.

➢ To minimize fatigue cracking, the stiffness value $G^* \times \sin(\delta)$ of the PAV binder must be less than 5000kPa at the intermediate pavement design temperature.

Table 5.1:- Binder characterisation under standard conditions of SHRP for VG-30 and VG-30 binder modified with various % of sulphur mixing at 100° C

BINDER CHARACTERISATION UNDER STANDARD CONDITIONS								
	TEST CONDITIONS		RESULTS OBTAINED				REMARKS	
Binder Type	Temperature °C	Angular Frequency rad/s	Phase Angle °	Strain	Complex Modulus Pa	G*/Sin(δ) Pa	Specifications Pa	Remarks
VG-30	60	10.03	85.82	0.1193	2252	2258	>1000	Ok
VG-30 +2% S	60	10.03	84.35	0.1203	3262	3278	>1000	Ok
VG-30 +3% S	60	10.03	85.14	0.1205	2728	2738	>1000	Ok
VG-30 +4% S	60	10.03	85.4	0.1213	2267	2274	>1000	Ok
VG-30 +6% S	60	10.03	86.03	0.1224	1586	1590	>1000	Ok
VG-30 +8% S	60	10.03	86.5	0.1249	1388	1390	>1000	Ok

From Table 5.1 it is observed that all the modified binder's complex modulus value satisfies the SHRP standard value. It shows that increase in complex modulus and decrease in phase angle of modified binder up to 4% sulphur by weight as compared to neat VG-30 bitumen. So it indicates higher resistance to deformation. The addition of sulphur in bitumen positively affects the rutting factor, thus, enhancing the rutting resistance of pavement mix. But addition more sulphur gradually reduces the complex modulus and G*/sin (δ) which indicates that the rutting resistance reduces with more sulphur addition. It also observed that the addition of 2% sulphur by weight indicates the highest value of complex modulus and lowest value of phage angle, which taken as the optimum modifier content.

5.2.1.2 Amplitude Sweep Test Results

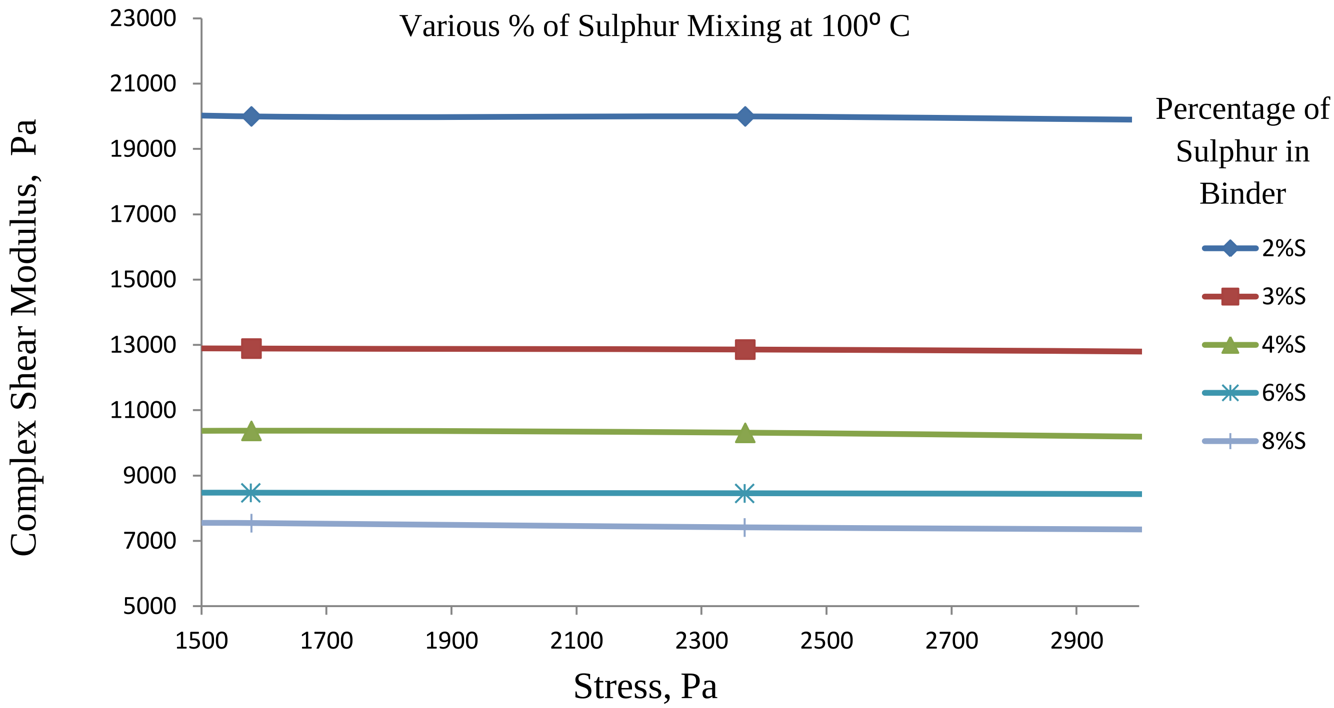

Figure 5.1 Complex Shear Modulus (G*) versus Stress

The results of amplitude sweep of various modified binder are analysed, to determine the linear viscoelastic region. Based on the viscoelastic region limits the input stress parameter for other rheological tests is chosen.

For all the modified binders, it has been found that the linear viscoelastic region range lies between 1500 Pa to 3000Pa. The same stress is applied for other tests. Therefore 2300Pa was chosen as the applied stress used as input applied stress parameter for other sweep tests.

5.2.1.3 Frequency Sweep Test Results

The maximum loading frequency (10 Hz) was selected because it intended loading in high traffic speed, and the minimum testing frequency (0.1 Hz) was taken because it intended loading in slow moving vehicle conditions.

The test results are compared with in the above mentioned range of frequencies for all binders and illustrated in following figures.

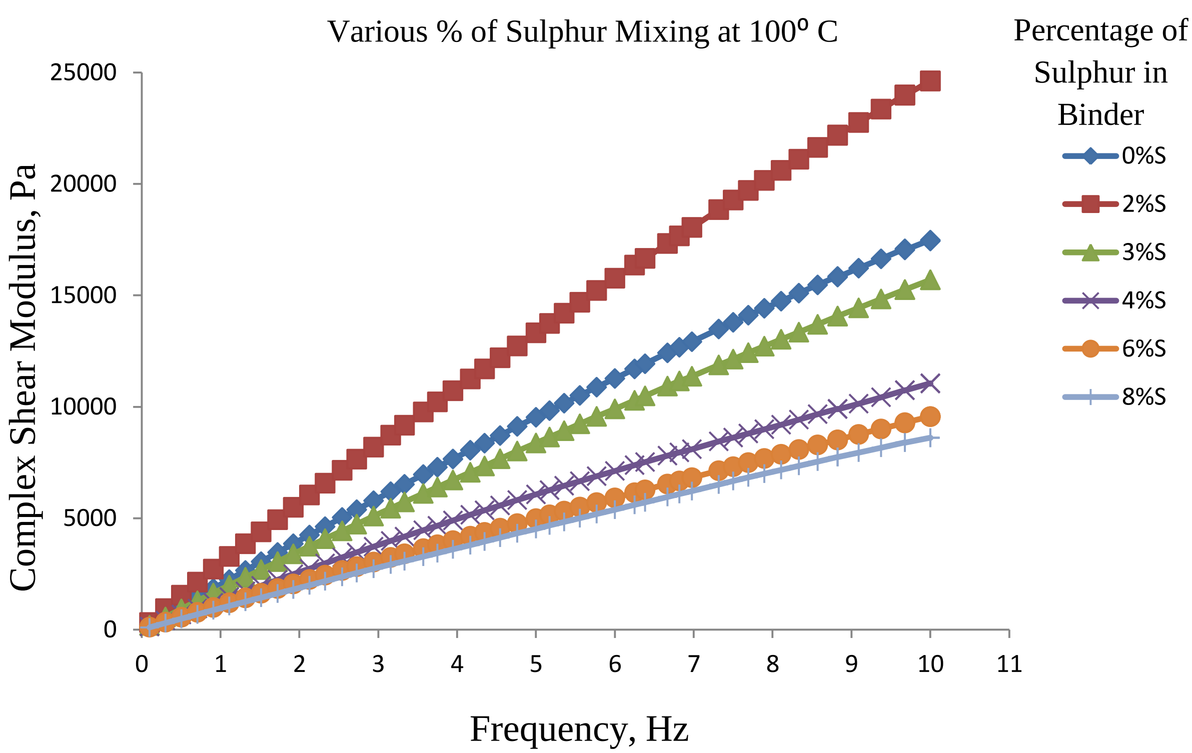

Figure 5.2 Complex Shear Modulus (G*) Versus Frequency

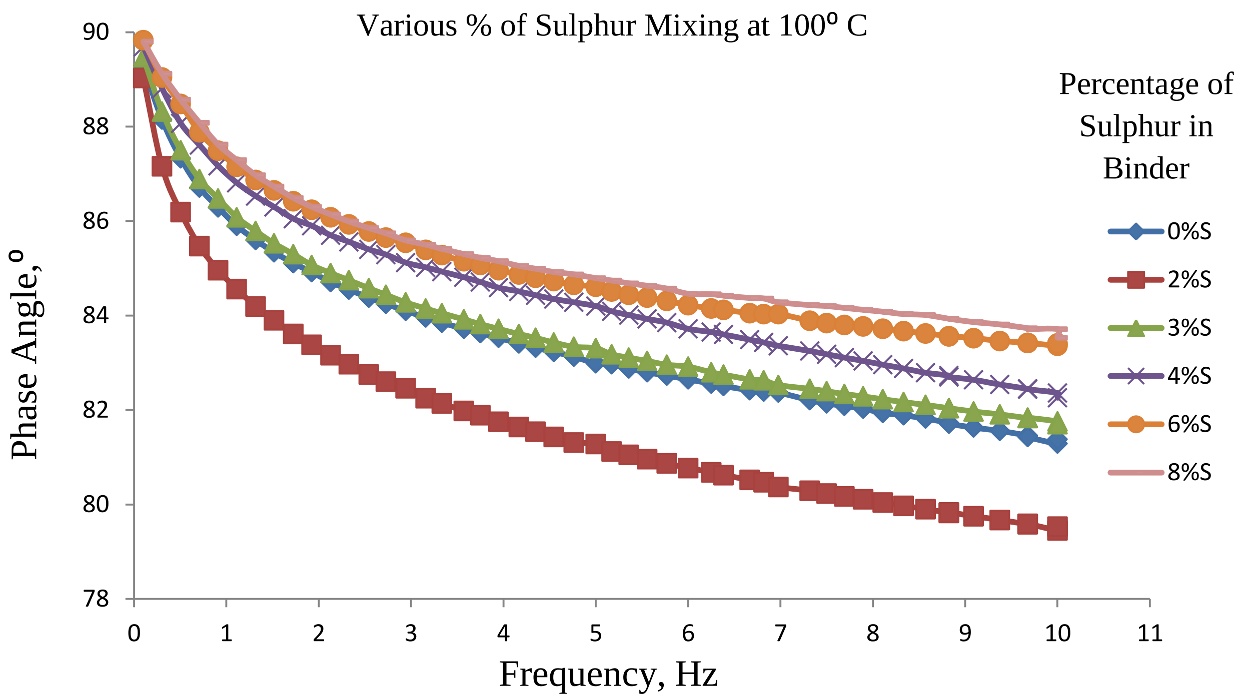

Figure 5.3 Phase Angle versus Frequency

It is clear from the above figures 5.2 and 5.3 that the general trend is that the complex shear modulus (G*) increases with the increase of frequencies. Also only 2% sulphur modified bitumen shows the greater complex modulus than the VG-30 neat bitumen. The general trend of phase angle is decreases with increase in frequencies. It appears clearly that the 2% sulphur modified bitumen shows less phase angle than neat bitumen, which directly affects the elastic properties. It indicates that the addition of sulphur in fewer amounts to the bitumen produces hard and elastic bitumen.

5.2.14 Temperature Sweep Test Results

Temperature has great effect on properties bitumen binder such as stiffness. The general properties of bituminous binder are at high temperature it behaves like fluid and its complex modulus is comparatively less.

The rheological properties in terms of phase angle and complex shear modulus are presented below in figure 5.4 and 5.5

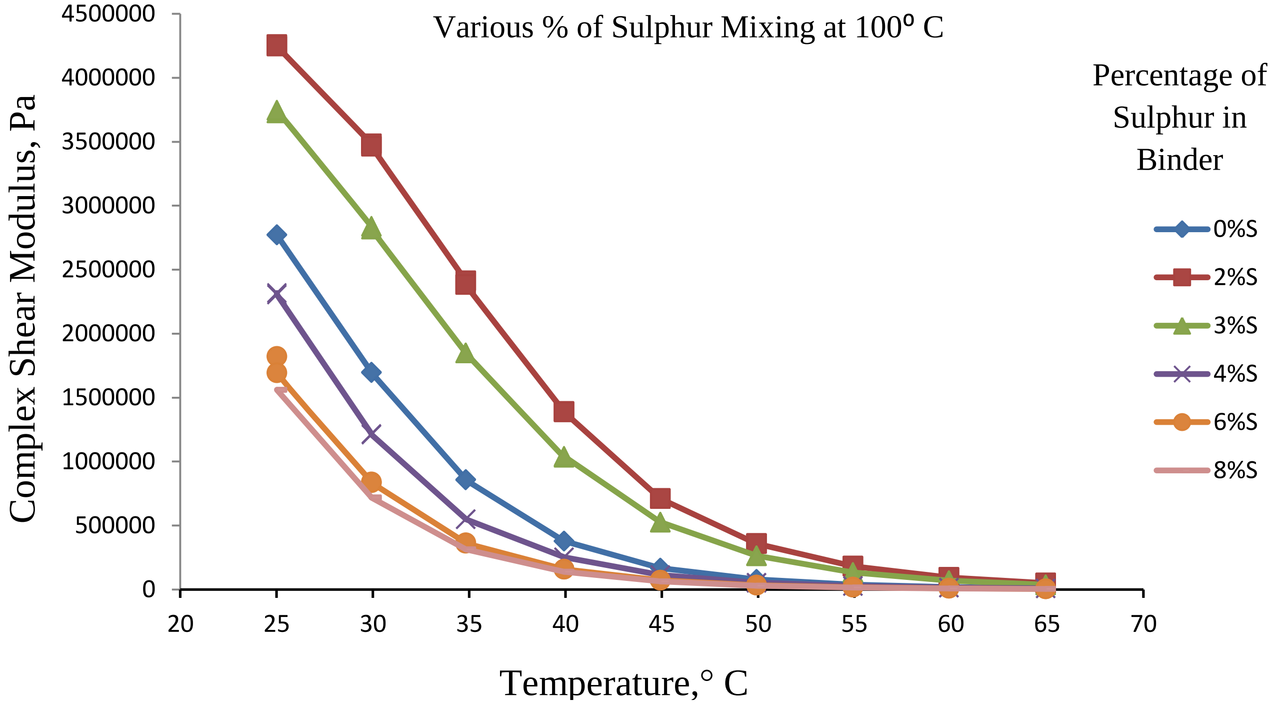

Figure 5.4 Complex Shear Modulus (G*) versus Temperature

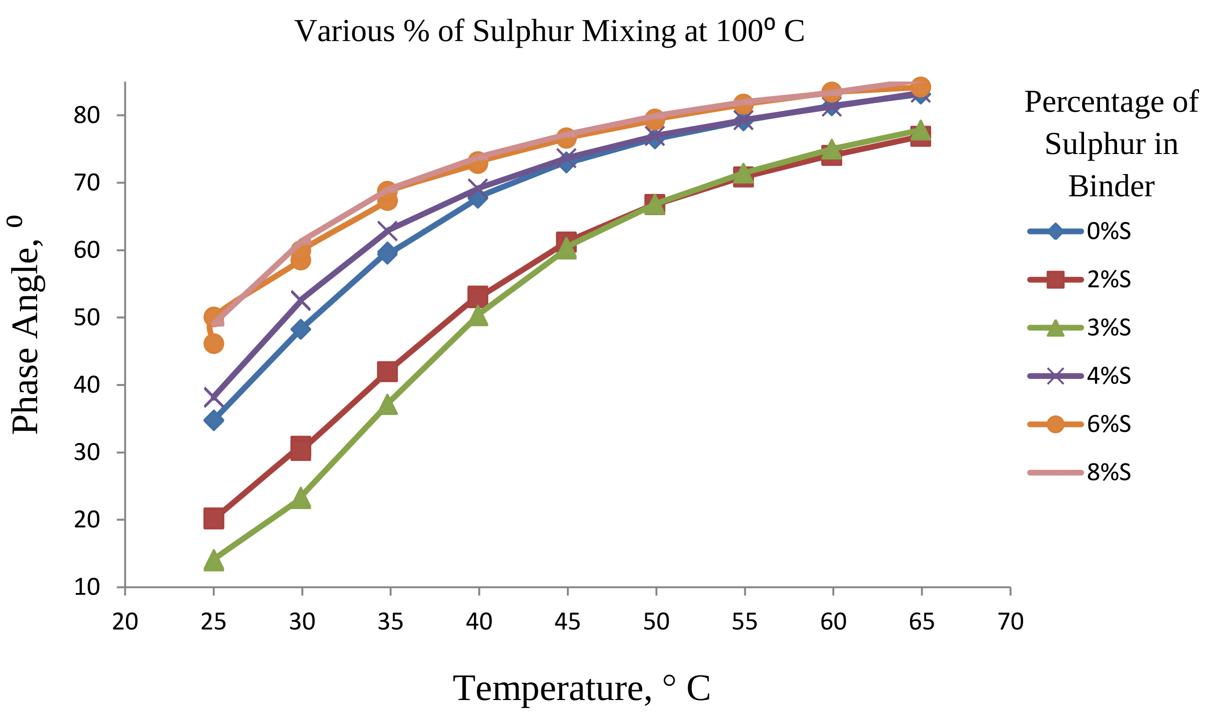

Figure 5.5 Phase Angle versus Temperature

From the results it is clear that when the temperature is increased, the rheological parameter complex shear modulus decreases. The bitumen modified with 2% sulphur and 3% sulphur shows higher complex modulus and less phase angle than the neat bitumen. This means that using sulphur as a bitumen modifier affects the rheological properties of bitumen as the modified binder improves its elasticity at high temperatures and also improves the flexibility at low temperature. Therefore it improves the resistance of rutting and fatigue cracking.

5.2.2 The Rheological Characterisation of Unaged Binder on Variation of Temperature

5.2.2.1Characterization of Binders under Standard Conditions of SHRP

Table 5.2 Binder characterisation under standard conditions of SHRP for VG-30 binder modified with 2% of sulphur at various mixing temperature

BINDER CHARACTERISATION UNDER STANDARD CONDITIONS								
	TEST CONDITIONS		RESULTS OBTAINED				REMARKS	
Binder Type	Temperature °C	Angular Frequency rad/s	Phase Angle °	Strain	Complex Modulus Pa	G*/Sin(δ) Pa	Specifications Pa	Remarks
VG30+2%S 95° C	60	10.03	85.27	0.1235	2720	2729	>1000	Ok
VG30+2%S 100° C	60	10.03	84.35	0.1224	3262	3278	>1000	Ok
VG30+ 2%S 105° C	60	10.03	85.56	0.1209	2686	2694	>1000	Ok
VG30+ 2%S 115° C	60	10.03	85.65	0.1206	2409	2416	>1000	Ok
VG30+ 2%S 125° C	60	10.03	85.72	0.1203	2332	2339	>1000	Ok
VG30+ 2%S 135° C	60	10.03	85.91	0.1200	2027	2027	>1000	Ok

The above table shows the characterisation of binder modified with 2% sulphur by weight but the mixing is prepared at various temperatures. It describes that all the variation satisfy the SHRP characterisation. It clears that the modified sulphur mixing at 95^{o}C the maximum stiffness. Therefore 95^{o} C blending temperature considered as the optimum blending temperature.

5.2.2.2 Amplitude Sweep Test Results

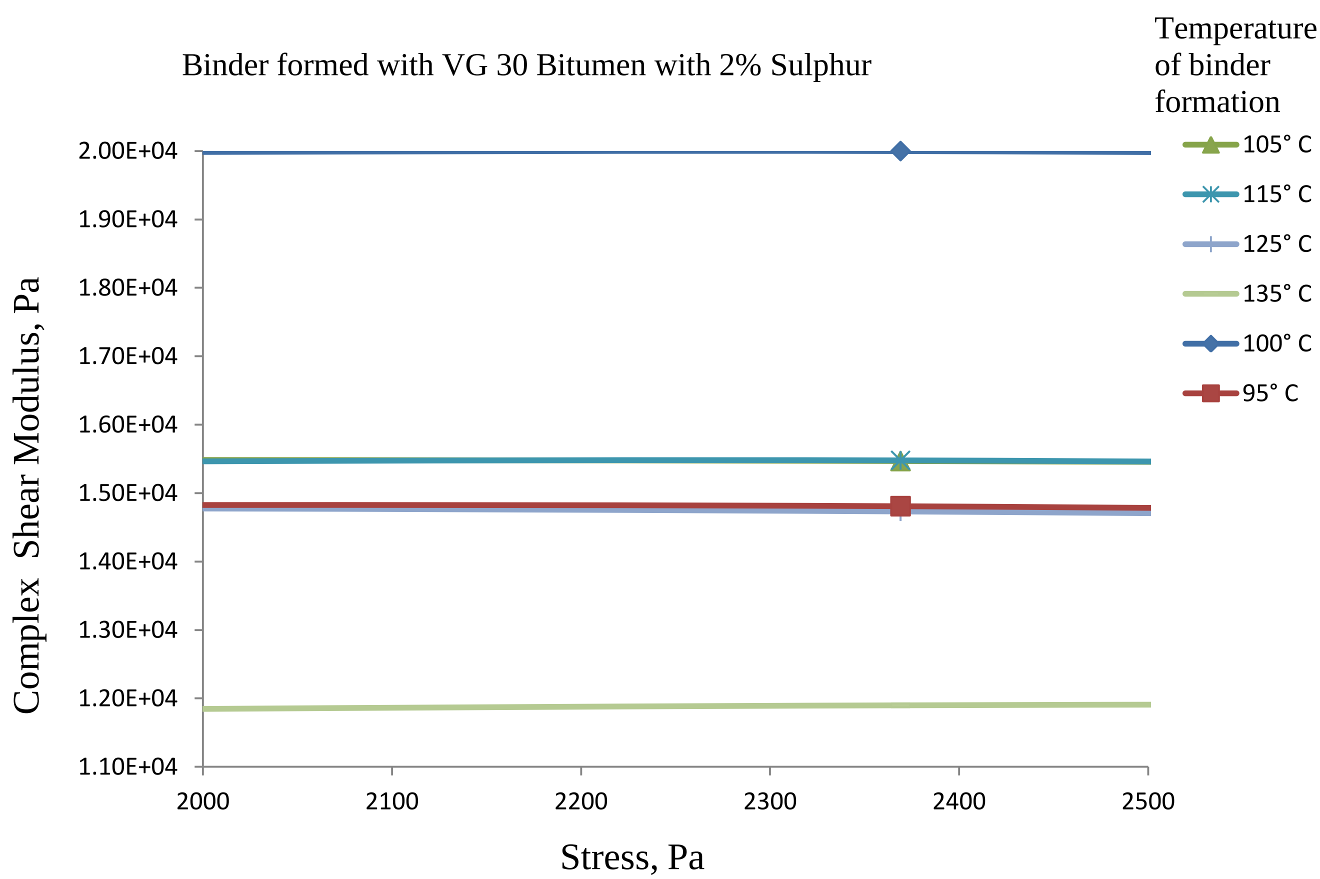

Figure 5.6 Complex Shear Modulus (G*) Versus Stress

The results of amplitude sweep of various modified binder are analysed, to determine the linear viscoelastic region. Based on the viscoelastic region limits the input stress parameter for further rheological tests is chosen.

For all the modified binders, it has been found that the linear viscoelastic region range lies between 2000 Pa to 2500Pa. The same stress is applied for other tests. Therefore 2300Pa was chosen as the applied stress used as input applied stress parameter for other sweep tests.

5.2.2.3 Frequency Sweep Test Results

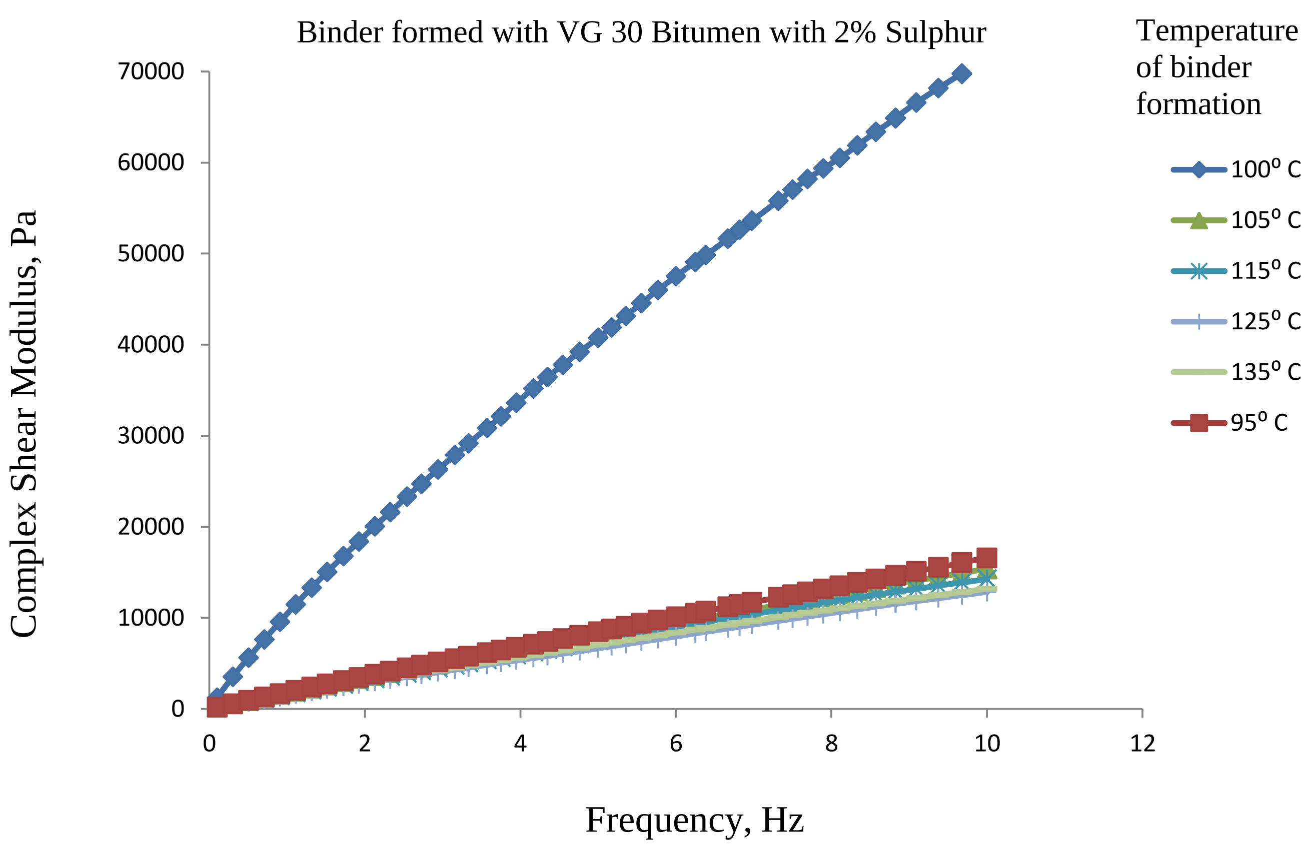

Figure 5.7 Complex Shear Modulus (G*) Versus Frequency

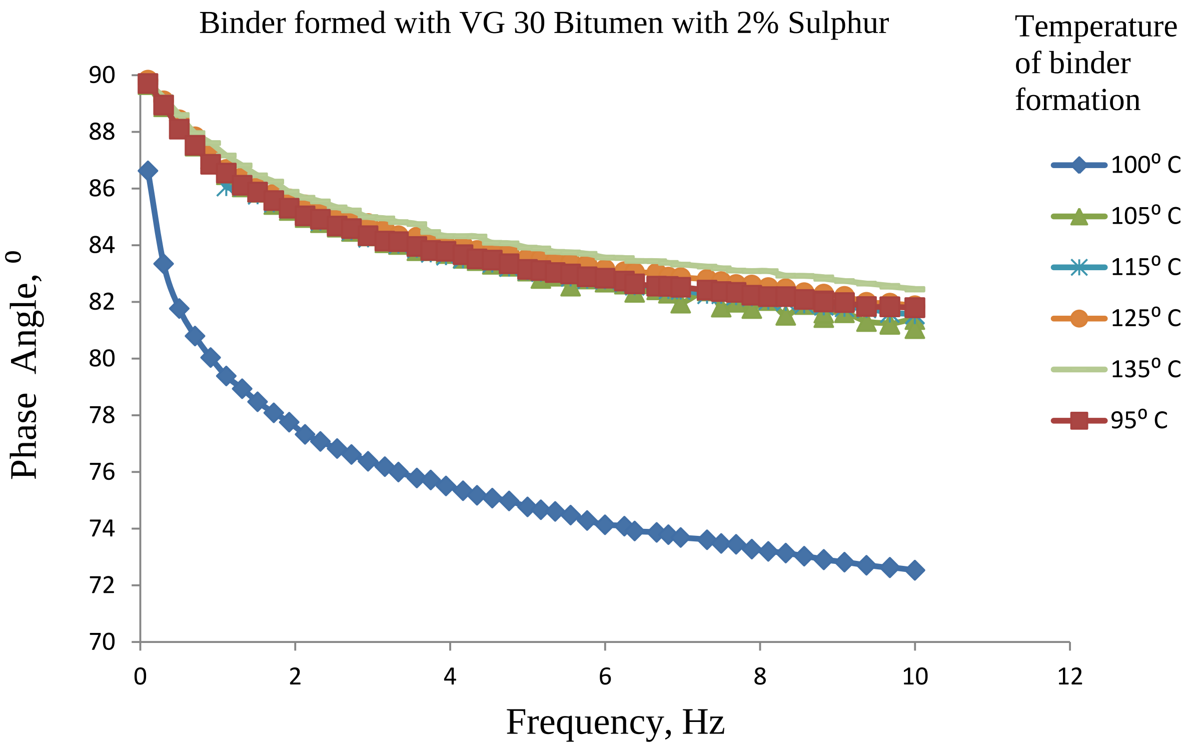

Figure 5.8 Phase Angle versus Frequency

The above figure describes that the general trend is maintained as complex shear modulus increases with increase in frequency and phase angle decrease with increase in frequency. Also it clears that except bitumen prepared at 95° C all others shows nearly similar characteristics. Therefore we can say that the blending temperature does not seriously affect the rheological characteristics.

5.2.2.3Temperature Sweep Test Results

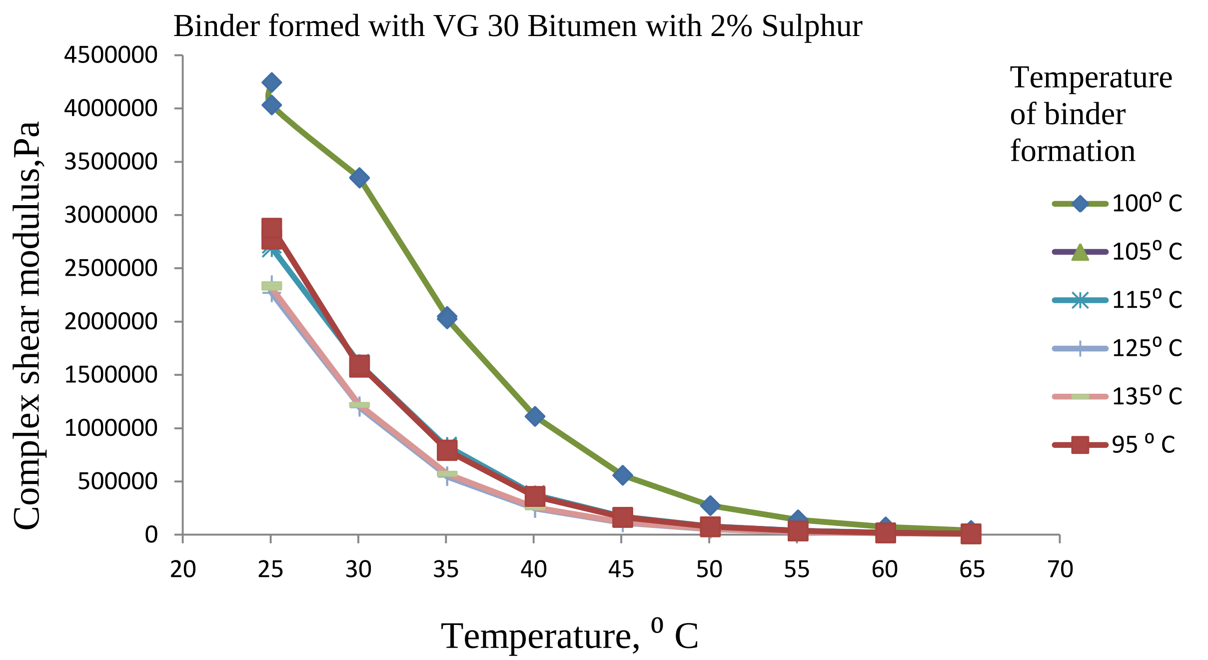

Figure 5.9 Complex Modulus versus Temperature

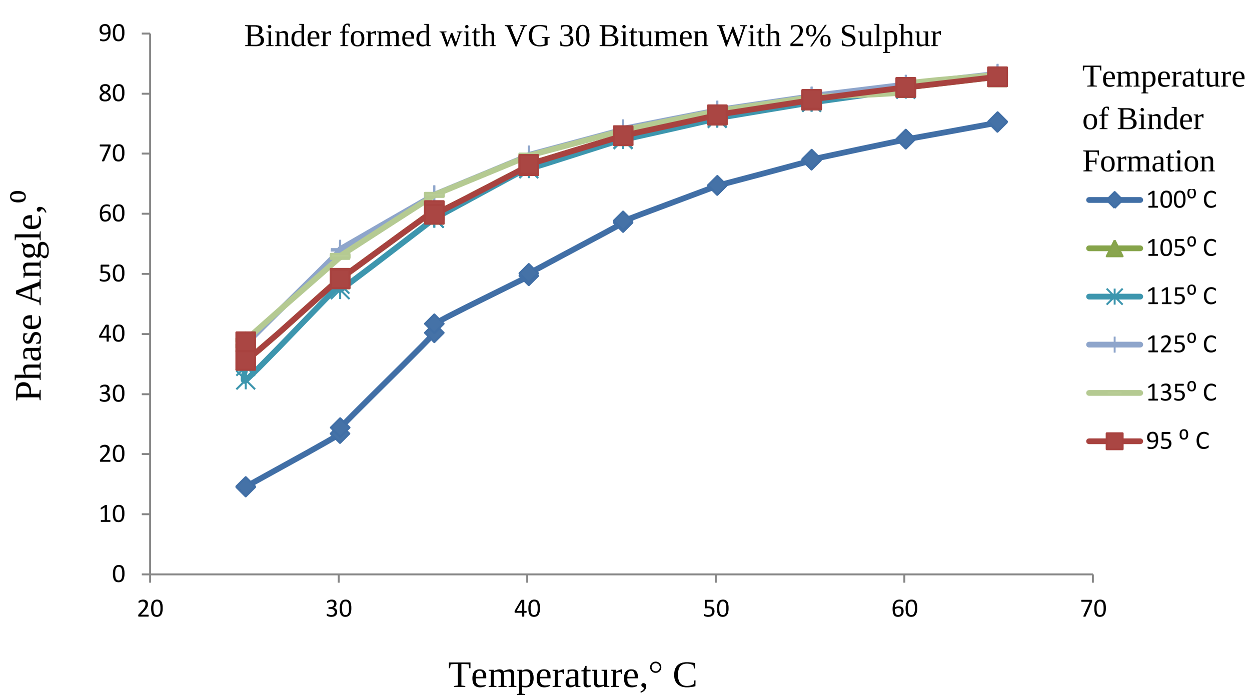

Figure 5.10 Phase Angle versus Temperature

From the results it is clear that when the temperature is increased, the rheological parameter complex shear modulus decreases. The bitumen modified with 2% sulphur at 95 ° C shows higher complex modulus and less phase angle than other mixing temperature. This means that using sulphur as a bitumen modifier affects the rheological properties of bitumen as the modified binder improves its elasticity at high temperatures and also improves the flexibility at low temperature. Therefore it improves the resistance of rutting and fatigue cracking

5.3 THE RHEOLOGICAL CHARACTERISATION OF RTFO AGED BINDER

5.3.1 The Rheological Characterisation of with various % of sulphur.

5.3.1.1 Characterisation of RTFO aged binder under standard condition oh SHRP

Table 5.3 it is observed that all the modified binder's complex modulus value satisfies the SHRP standard value. It shows that increase in complex modulus and decrease in phase angle of modified binder up to 3% sulphur by weight as compared to RTFO aged VG-30 bitumen. So it indicates higher resistance to deformation. It also observed that the addition of 2% sulphur by weight indicates the highest value of complex modulus and lowest value of phage angle, which taken as the optimum modifier content

Table 5.3 Binder characterisation under standard conditions of SHRP for RTFO aged binder (VG-30 and VG-30 binder modified with various % of sulphur mixing at 100° C)

BINDER CHARACTERISATION UNDER STANDARD CONDITIONS								
	TEST CONDITIONS		RESULTS OBTAINED				REMARKS	
Binder Type	Temperature °C	Angular Frequency rad/s	Phase Angle °	Strain	Complex Modulus Pa	G*/Sin(δ) Pa	Specifications Pa	Remarks
RTFO aged VG-30	60	10.03	76.72	0.0108	5507	5549	>2200	Ok
RTFO aged (VG-30 +2% S)	60	10.03	72.73	0.0077	2.32E+04	2.43E+04	>2200	Ok
RTFO aged (VG-30 +3% S)	60	10.03	75.45	0.008	2.25E+04	2.32E+04	>2200	Ok
RTFO aged (V G-30 +4% S)	60	10.03	78.76	0.0174	1.03E+04	1.05E+04	>2200	Ok
RTFO aged (VG-30 +6% S)	60	10.03	79.38	0.0205	8775	8901	>2200	Ok
RTFO aged (VG-30 +8% S)	60	10.03	80.33	0.0229	7866	8013	>2200	Ok

5.3.1.2 Amplitude Sweep Test Result

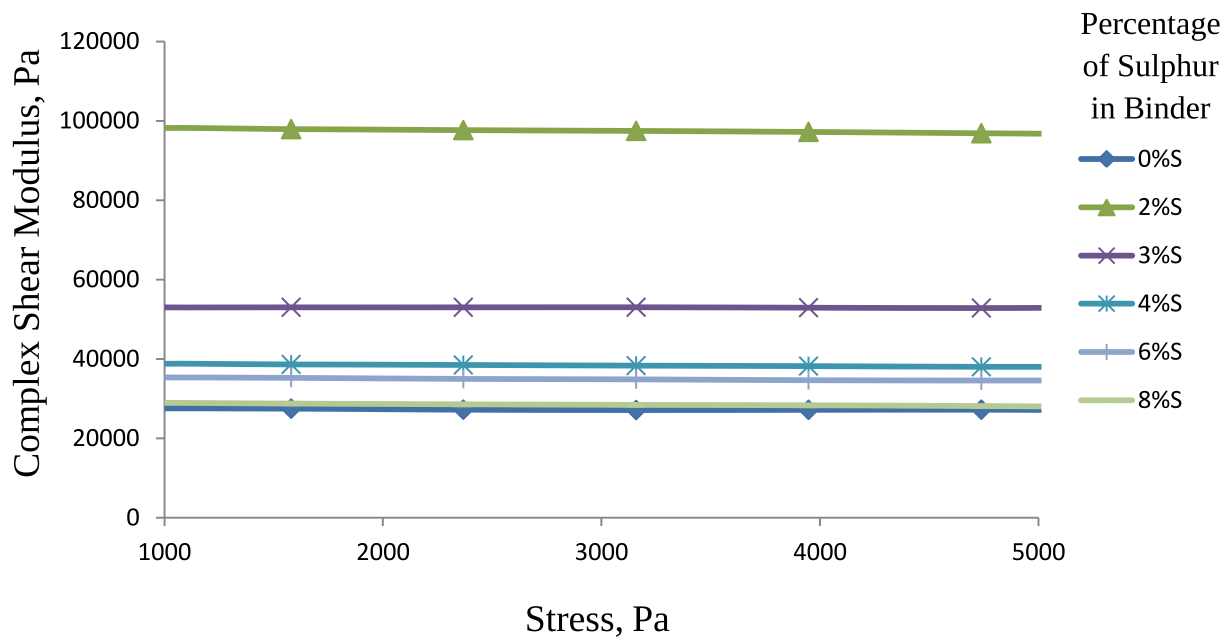

Figure 5.11 Complex Modulus versus Stress

The results of amplitude sweep of various modified binder are analysed, to determine the linear viscoelastic region. Based on the viscoelastic region limits the input stress parameter for other rheological tests is chosen.

For all the modified binders, it has been found that the linear viscoelastic region range lies between 1000 Pa to 5000Pa. The same stress is applied for other tests. Therefore 3000Pa was chosen as the applied stress used as input applied stress parameter for other sweep tests.

5.3.1.3 Frequency Sweep Test Result

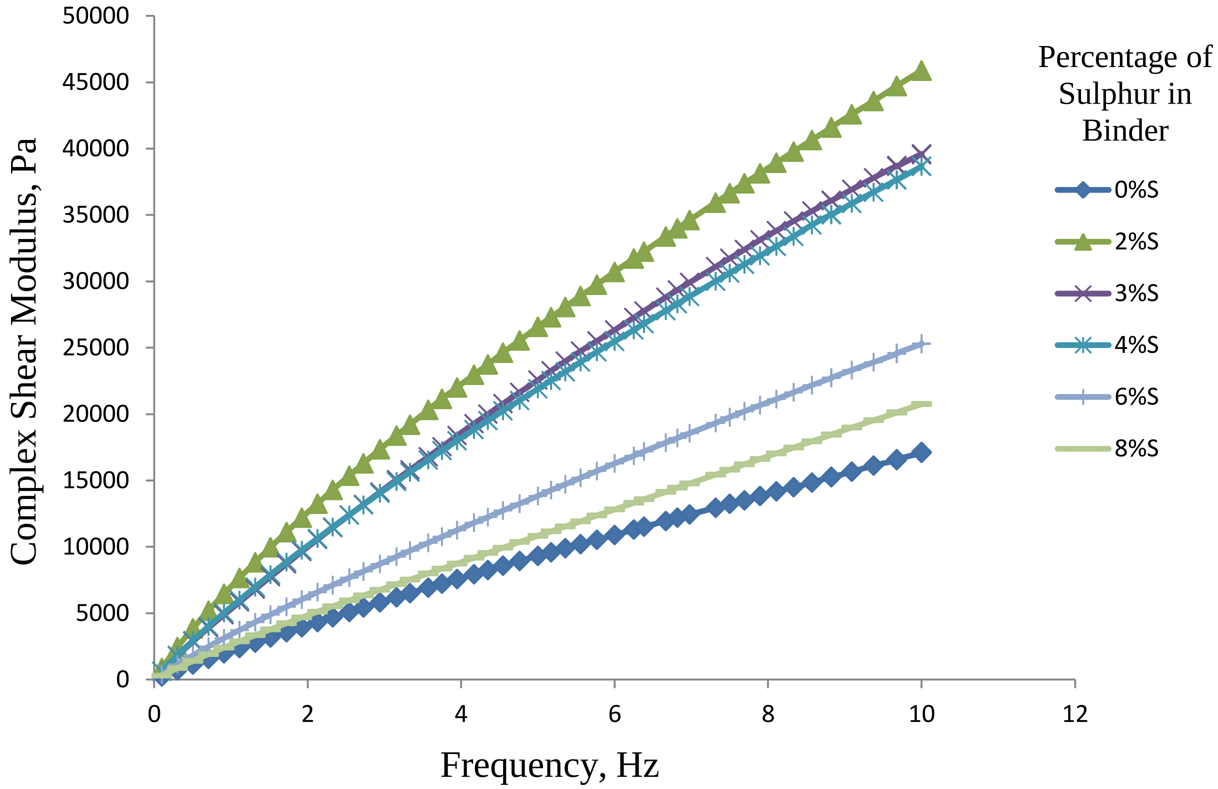

Figure 5.12 Complex Modulus versus Frequency

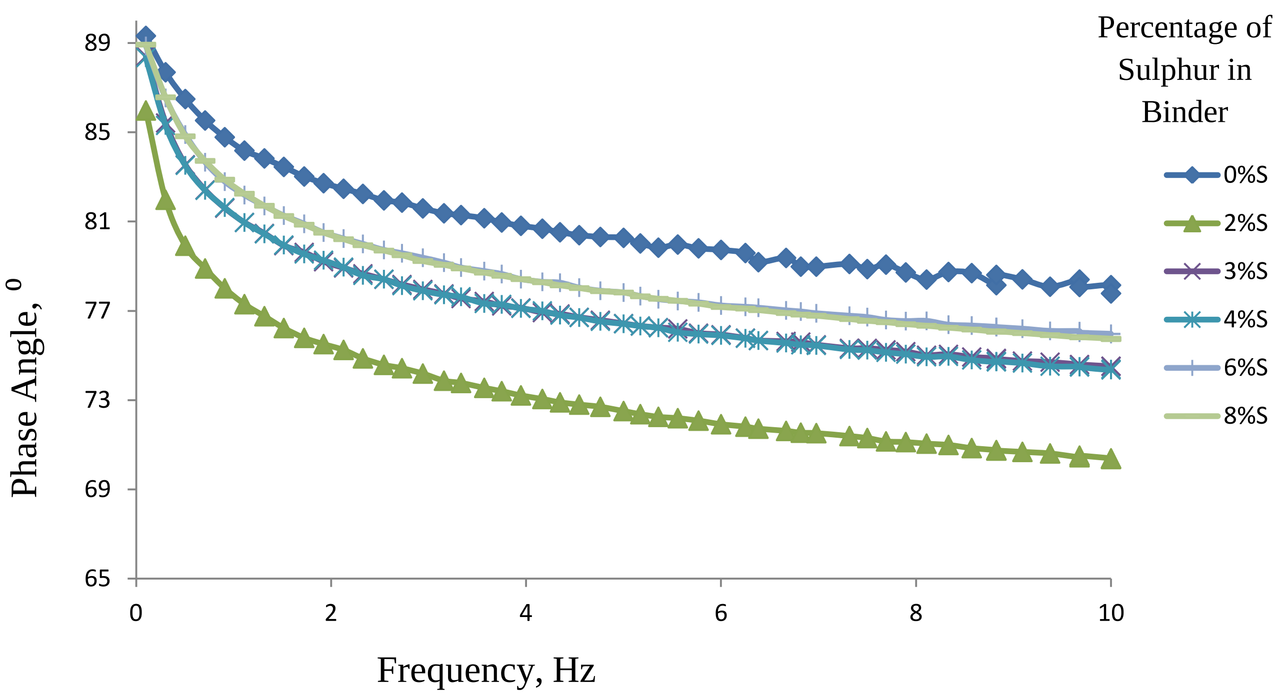

Figure 5.13 Phase Angle versus Frequency

It is clear from the above graphs 5.12 and 5.13 that the general trend is that the complex shear modulus (G*) increases with the increase of frequencies. Also only 2% sulphur modified RTFO aged bitumen shows the greater complex modulus than the RTFO aged VG-30 bitumen. The general trend of phase angle is decreases with increase in frequencies. It appears clearly that the 2% sulphur modified bitumen shows less phase angle than neat bitumen, which directly affects the elastic properties. It describes that all the RTFO aged modified binder has improved rheological property than RTFO aged VG-30 bitumen..

5.3.1.4 Temperature Sweep Test Result

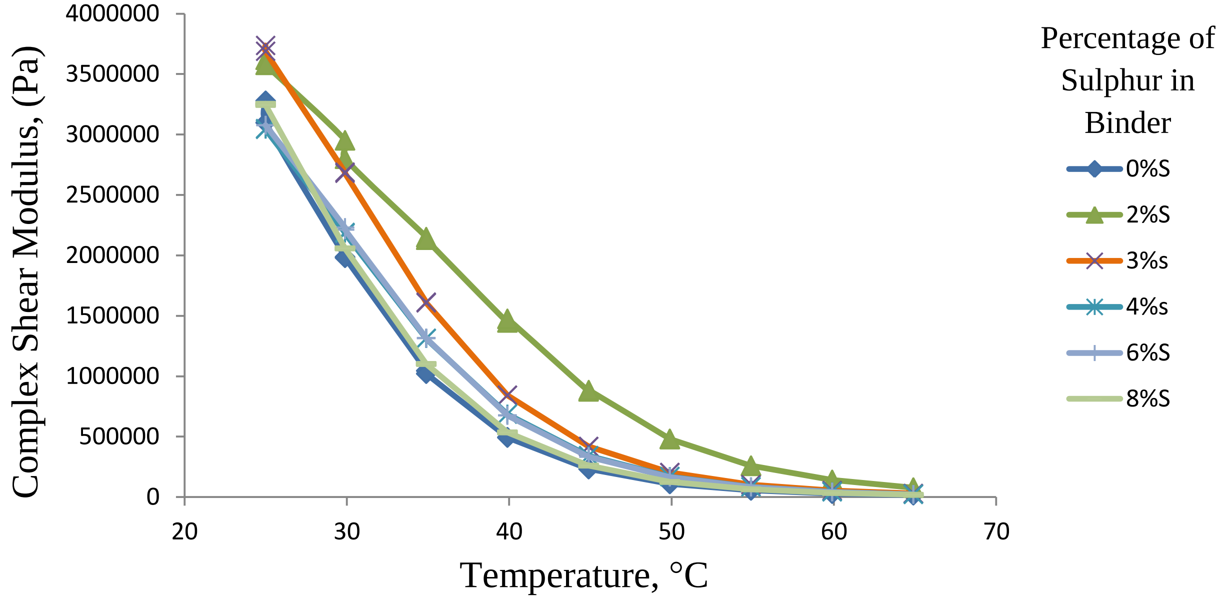

Figure 5.14 Complex Modulus versus Temperature

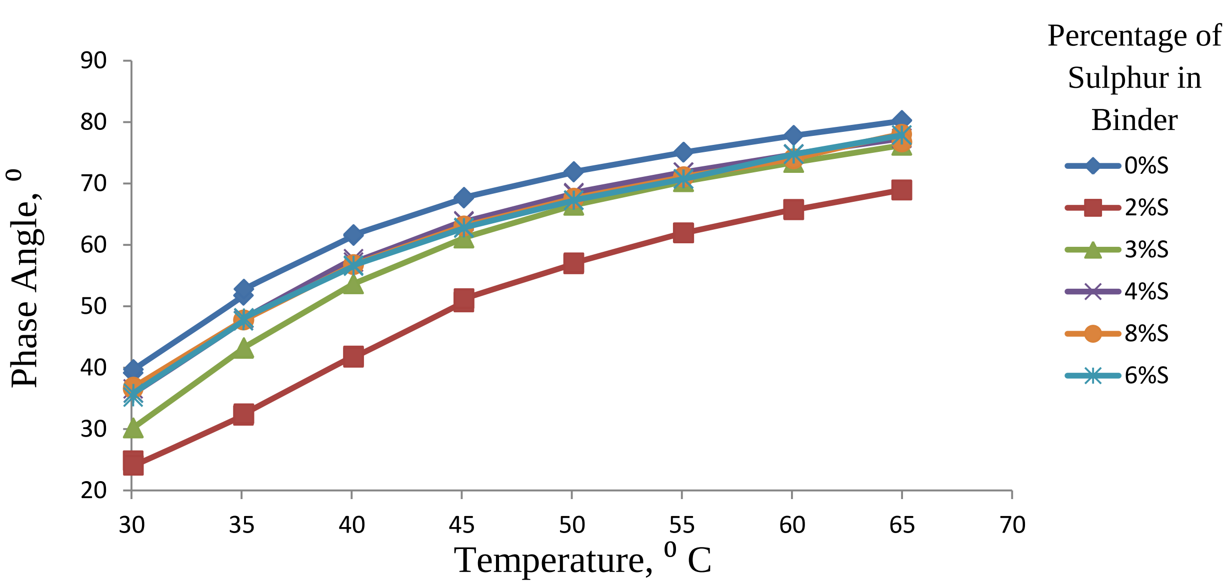

Figure 5.15 Phase Angle versus Temperature

The complex modulus curves show the same general behaviour for TFOT conditions. The RTFO materials were obviously stiffer than the unaged samples over the entire range of frequencies which indicates that once the binder is aged sufficiently the stiffness changes greatly with the frequency loading.

Also the phase angles for RTFO aged materials were much lower than that of the unaged samples. Hence it is concluded that as the binder is aged it becomes stiffer and more elastic.

5.3.2 The Rheological Characterisation of RTFO Aged Binder on Variation of Temperature

5.3.2.1 Characterization of RTFO Aged Binder under Standard Condition Of SHRP

The Table 5.4 presented below and from it observed that all the modified binder's complex modulus value satisfies the SHRP standard value. It shows that the bitumen mixing at 95^{o} C and 105^{o} C have much more value of complex modulus than other mixing temperature. So it indicates higher resistance to deformation. It also observed that the addition of 2% sulphur by weight mixing at 95^{o} C shows the highest value of complex modulus and lowest value of phage angle.

Table 5.4 Binder characterisation under standard conditions of SHRP for RTFO aged (VG-30 modified with 2% of sulphur at various mixing temperature)

BINDER CHARACTERISATION UNDER STANDARD CONDITIONS								
	TEST CONDITIONS		RESULTS OBTAINED				REMARKS	
Binder Type	Temperature °C	Angular Frequency rad/s	Phase Angle °	Strain	Complex Modulus Pa	G*/Sin(δ) Pa	Specifications Pa	Remarks
RTFO aged VG30+2%S 95° C	60	10.03	80.62	0.024	7262	7360	>2200	Ok
RTFO aged VG30+2%S 100° C	60	10.03	72.73	0.0077	2.32E+04	2.43E+04	>2200	Ok
RTFO aged VG30+2%S 105° C	60	10.03	78.81	0.0143	1.26E+04	1.28E+04	>2200	Ok
RTFO aged VG30+2%S 115° C	60	10.03	80.19	0.0241	7464	7575	>2200	Ok
RTFO aged VG30+2%S 125° C	60	10.03	81.3	0.0250	7195	7277	>2200	Ok
RTFO aged VG30+2%S 135° C	60	10.03	81.54	0.0263	6843	6918	>2200	Ok

5.3.2.2 Amplitude Sweep Test Results

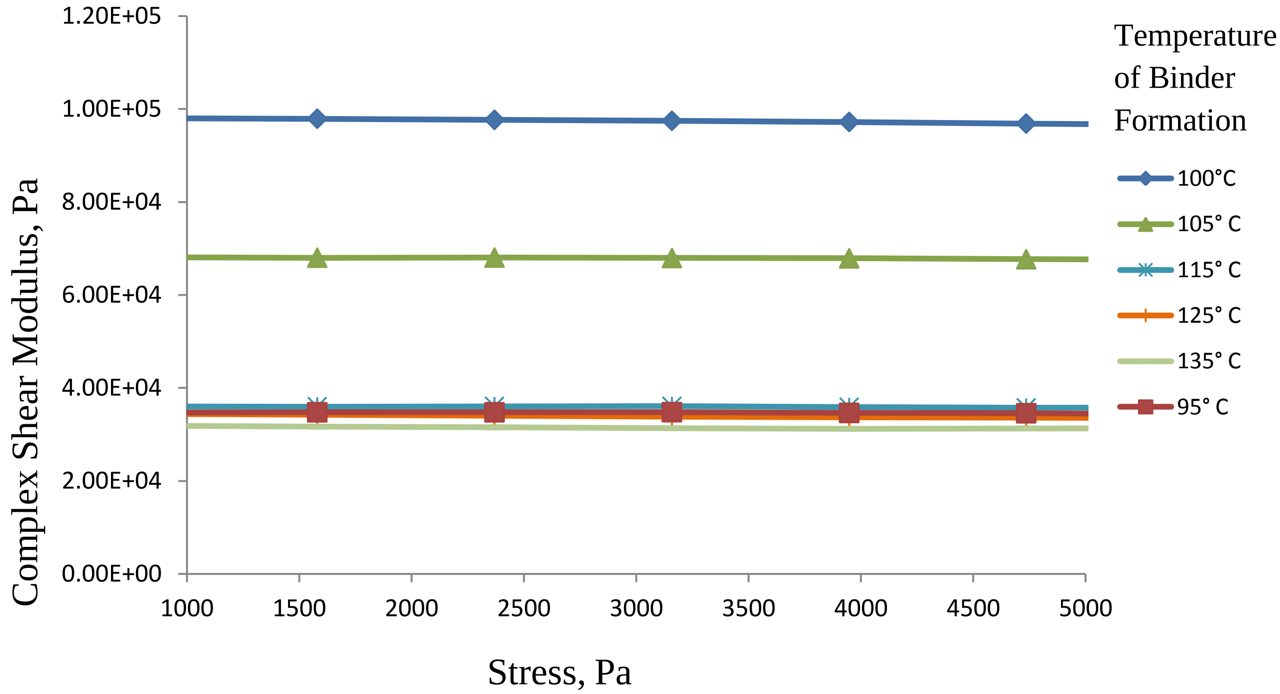

Figure 5.16 Complex Shear Modulus versus Stress

The results of amplitude sweep of various modified binder are analysed, to determine the linear viscoelastic region. Based on the viscoelastic region limits the input stress parameter for other rheological tests is chosen.

For all the modified binders, it has been found that the linear viscoelastic region range lies between 1000 Pa to 5000Pa. The same stress is applied for other tests. Therefore 3000Pa was chosen as the applied stress used as input applied stress parameter for other sweep tests.

5.3.2.3 Frequency sweep test results

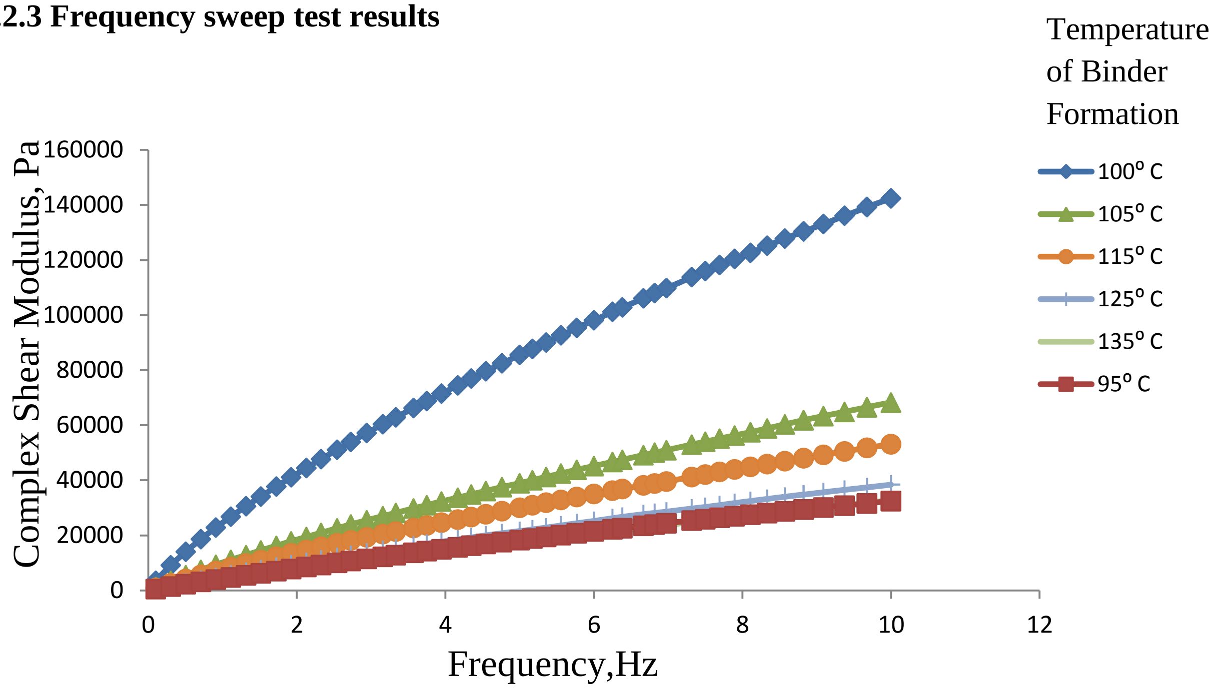

Figure 5.17 Complex Shear Modulus versus Stress

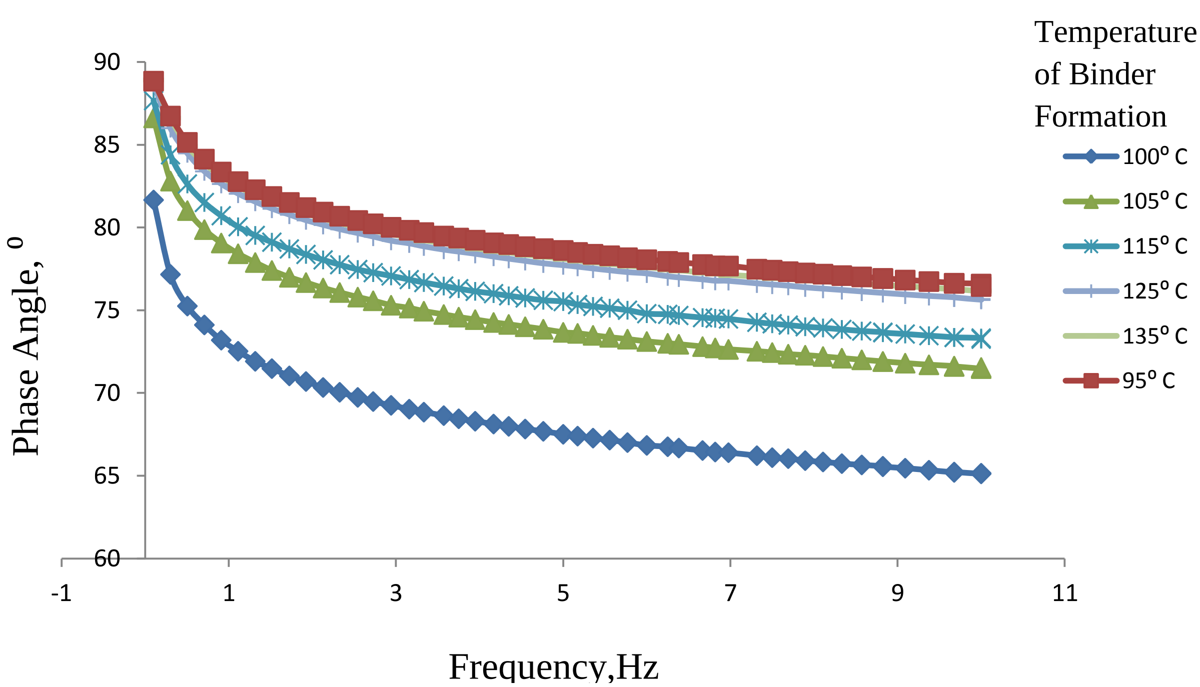

Figure 5.18 Phase Angle versus Stress

5.3.2.4 Temperature sweep test results

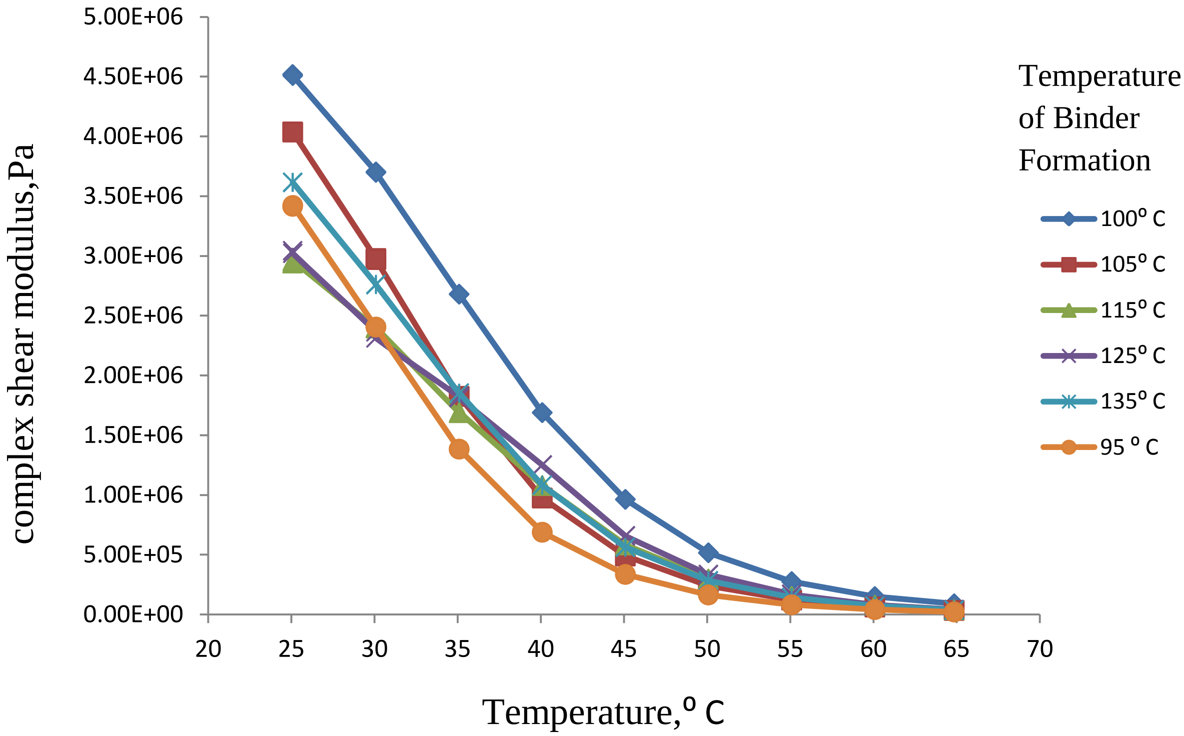

Figure 5.19 Complex shear modulus versus Temperature

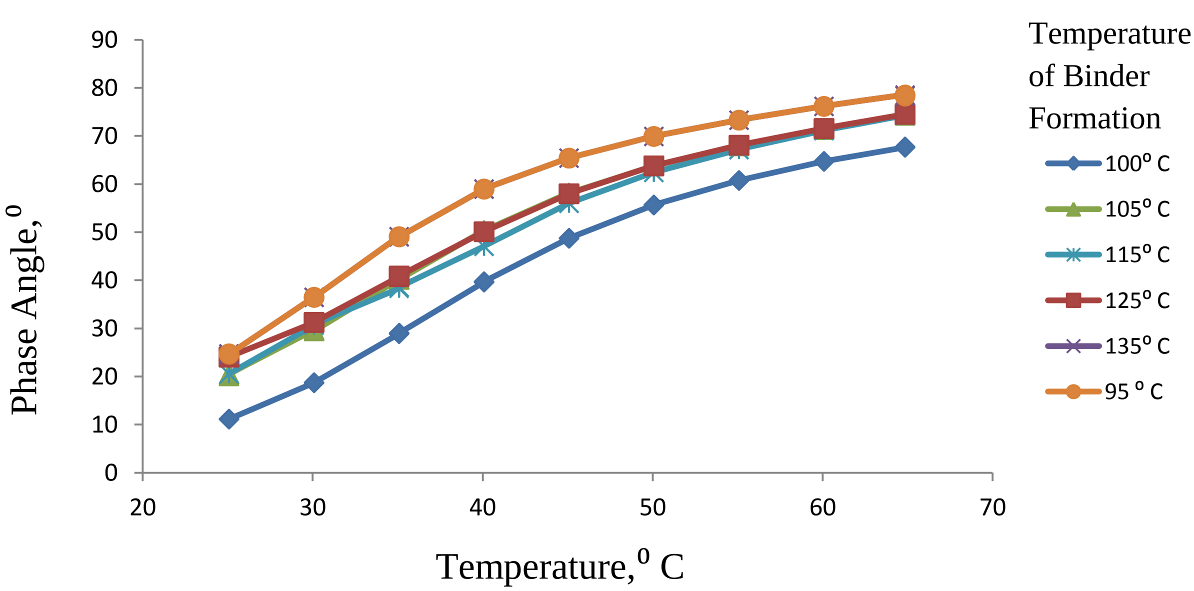

Figure 5.20 Phase Angle versus Temperature

5.4 THE RHEOLOGICAL CHARACTERISATION OF PAV AGED BINDER

5.4.1 Characterization of PAV aged binder under standard condition of SHRP on variation % of sulphur

5.4.1.1

Table 5.5 Binder characterisation under standard conditions of SHRP for PAV aged binder (VG-30 and VG-30 binder modified with various % of sulphur mixing at 100° C)

BINDER CHARACTERISATION UNDER STANDARD CONDITIONS								
	TEST CONDITIONS		RESULTS OBTAINED				REMARKS	
Binder Type	Temperature °C	Angular Frequency rad/s	Phase Angle °	Strain	Complex Modulus Pa	G*xSin(δ) Pa	Specifications kPa	Remarks
PAV aged VG-30	40	10.03	52.16	0.0719	7.00E+05	5.53E+05	<5000 kPa	Ok
PAV aged VG-30+2% S	40	10.03	40.75	0.0618	1.51E+06	9.86E+05	<5000 kPa	Ok
PAV aged VG-30 +3% S	40	10.03	49.71	0.0715	8.13E+05	6.54E+05	<5000 kPa	Ok
PAV aged VG-30 +4% S	40	10.03	51.5	0.0332	8.07E+05	6.37E+05	<5000 kPa	Ok
PAV aged VG-30 +6% S	40	10.03	54.4	0.0638	7.04E+05	5.73E+05	<5000 kPa	Ok
PAV aged VG-30 +8% S	40	10.03	56.1	2.426	6.14E+05	5.05E+05	<5000 kPa	Ok

From above table it clearly observed that all the aged binder satisfy the SHRP condition i.e. the G*x sin(δ) stiffness value for fatigue cracking is greater than 5000kPa. Also the 2% sulphur modified bitumen shows the highest stiffness value.

5.4.1.2 Amplitude Sweep Test Results

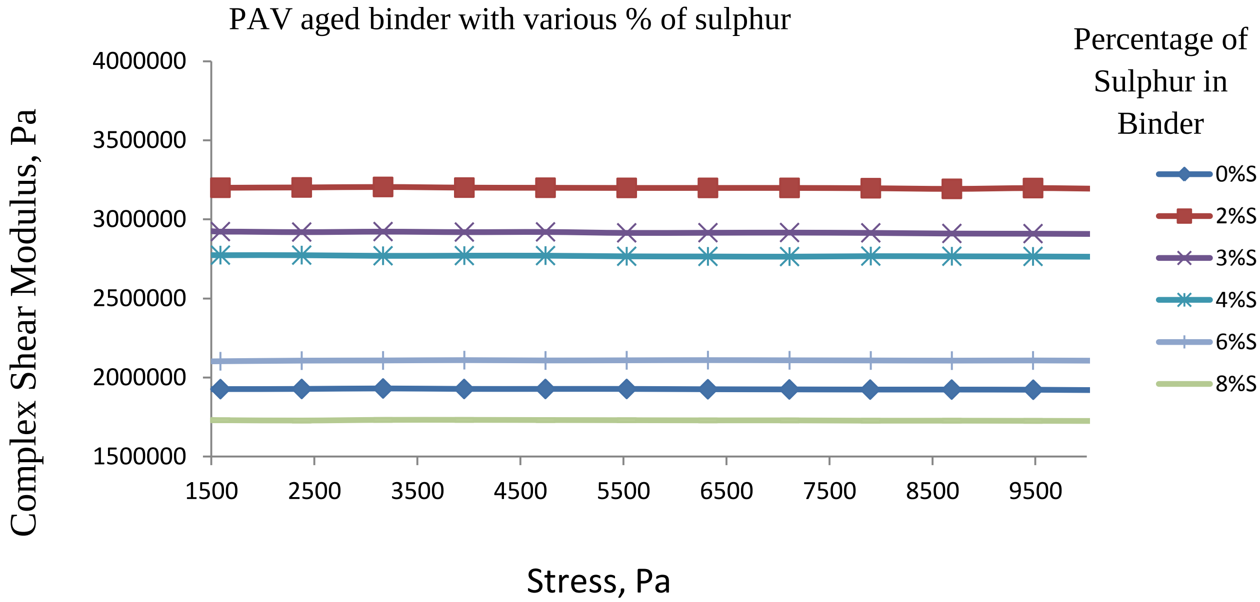

Figure 5.21 Complex Shear Modulus versus Stress

The results of amplitude sweep of various modified binder are analysed, to determine the linear viscoelastic region. Based on the viscoelastic region limits the input stress parameter for other rheological tests is chosen.

For all the modified binders, it has been found that the linear viscoelastic region range lies between 15000 Pa to 9500Pa. The same stress is applied for other tests. Therefore 5000Pa was chosen as the applied stress used as input applied stress parameter for other sweep tests.

5.4.1.3 Frequency sweep test results

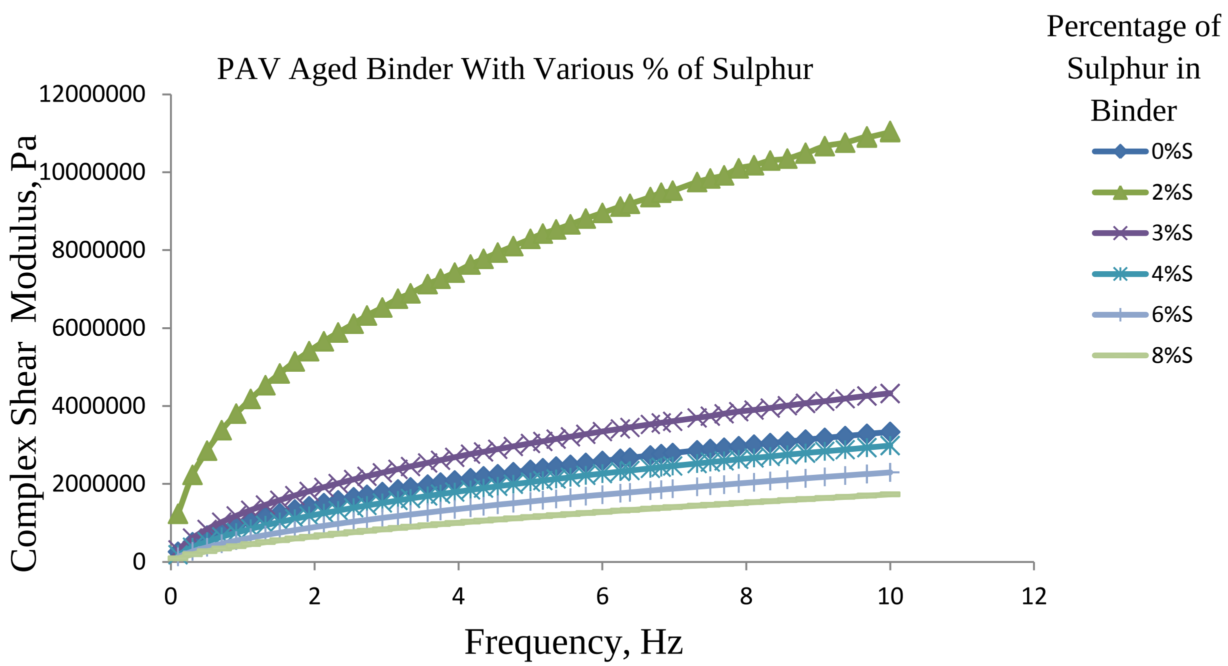

Figure 5.22 Complex Shear Modulus versus Frequency

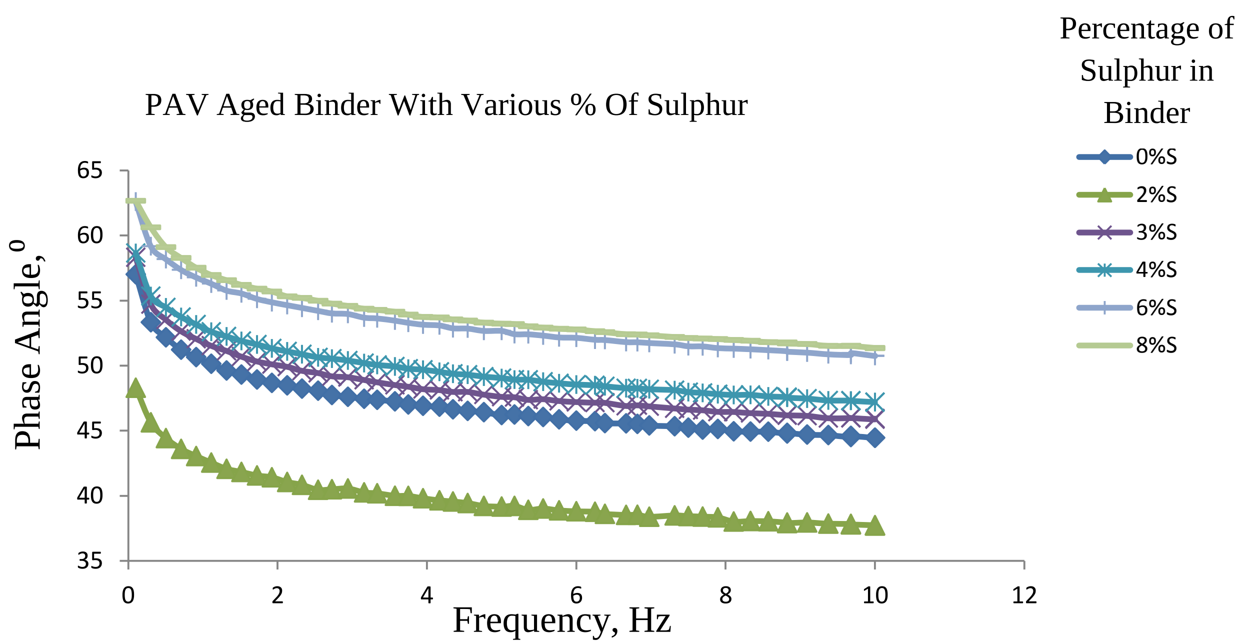

Figure 5.23 Phase Angle versus Frequency

5.4.1.4 Temperature Sweep Test Results

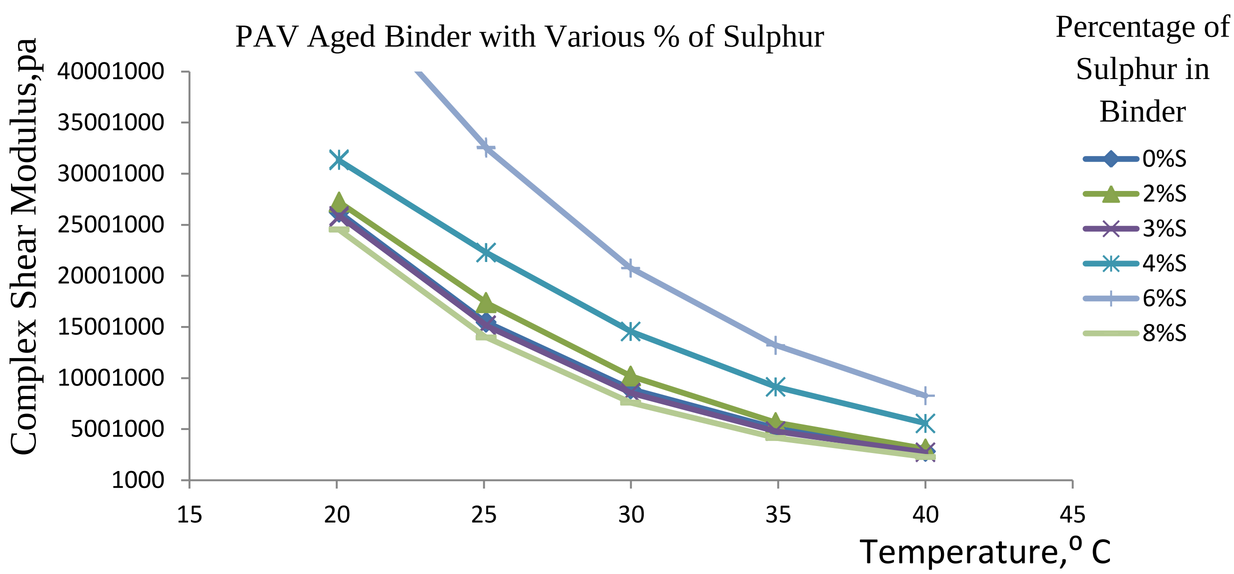

Figure 5.24 Complex Modulus versus Temperature

The above graph shows the general trend that complex modulus decreases with increase in temperature

5.4.2 The rheological characterisation of PAV aged binder on variation of temperature

5.4.2.1 Characterization of PAV aged binder under standard condition of SHRP

Table 5.6 Binder characterisation under standard conditions of SHRP for PAV aged binder (VG-30 and VG-30 binder modified with various temperatures)

BINDER CHARACTERISATION UNDER STANDARD CONDITIONS								
	TEST CONDITIONS		RESULTS OBTAINED				REMARKS	
Binder Type	Temperature °C	Angular Frequency rad/s	Phase Angle °	Strain	Complex Modulus Pa	G*/Sin(δ) Pa	Specifications kPa	Remarks
PAV aged VG30+2%S 95° C	40	10.03	59.92	0.2469	2.07E+05	1.79E+05	<5000 kPa	Ok
PAV aged VG30+2%S 100° C	40	10.03	49.96	0.03922	1.28E+06	9.80E+05	<5000 kPa	Ok
PAV aged VG30+2%S 105° C	40	10.03	52.14	0.05721	8.79E+05	6.94E+05	<5000 kPa	Ok
PAV aged VG30+2%S 115° C	40	10.03	59.11	0.1137	4.44E+05	3.85E+05	<5000 kPa	Ok
PAV aged VG30+2%S 125° C	40	10.03	60.17	0.1236	4.09E+05	3.51E+05	<5000 kPa	Ok
PAV aged VG30+2%S 135° C	40	10.03	60.5	0.1471	3.44E+05	2.99E+05	<5000 kPa	Ok

5.4.2.1 Amplitude sweep test results

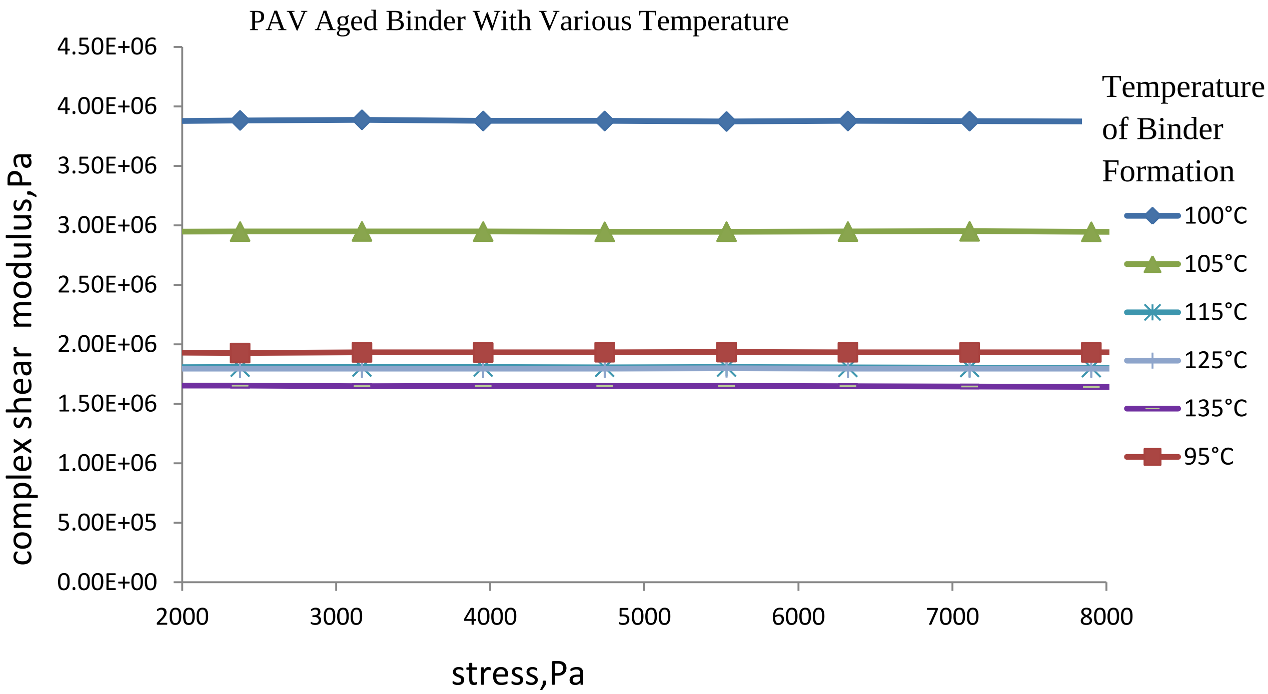

Figure 5.25 Complex Modulus versus Stress

5.4.2.2 Frequency sweep test results

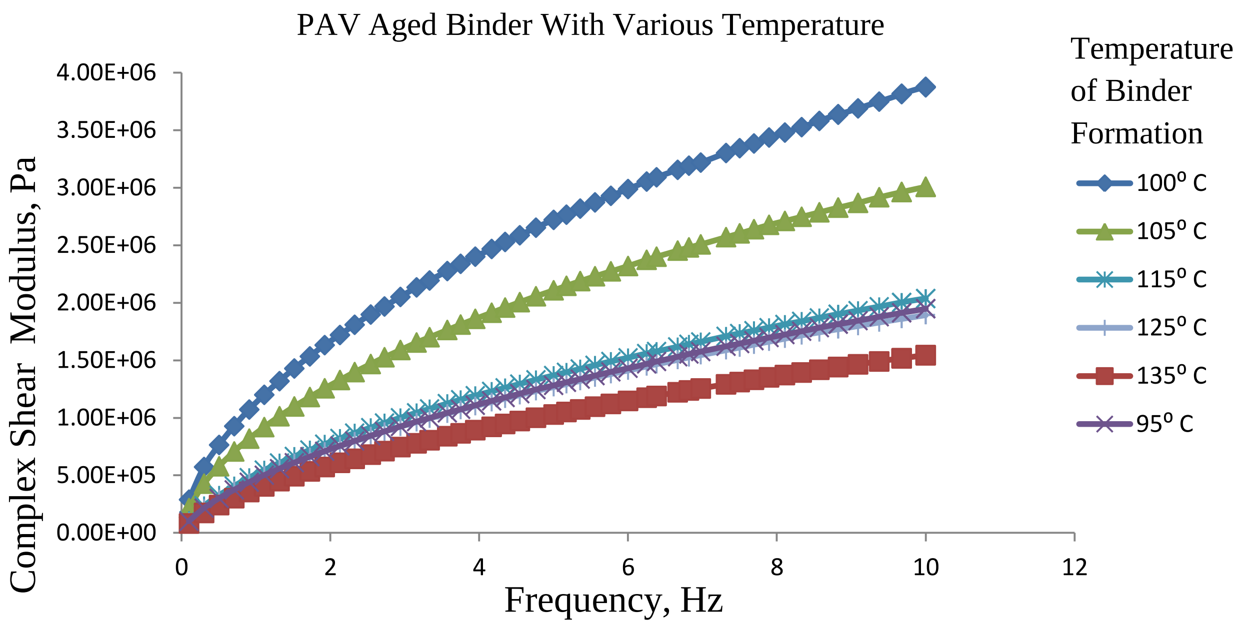

Figure 5.26 Complex Modulus versus Frequency

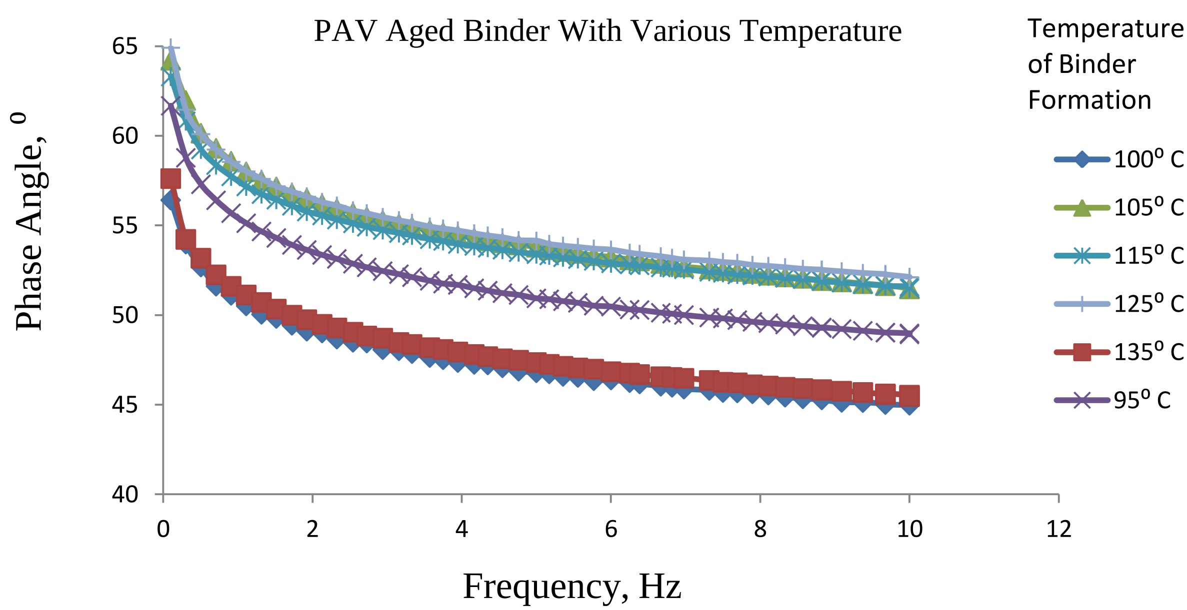

Figure 5.27 Phase Angle versus Frequency

5.4.2. Temperature Sweep Test Results

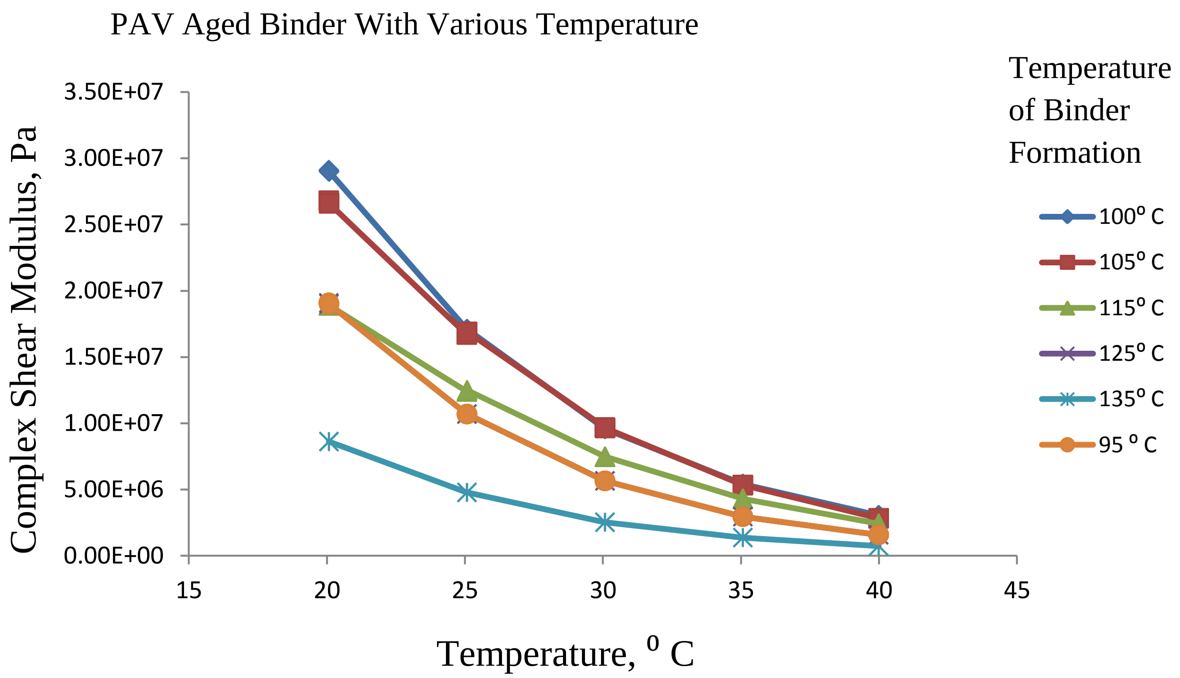

Figure 5.28 Complex Modulus versus Temperature

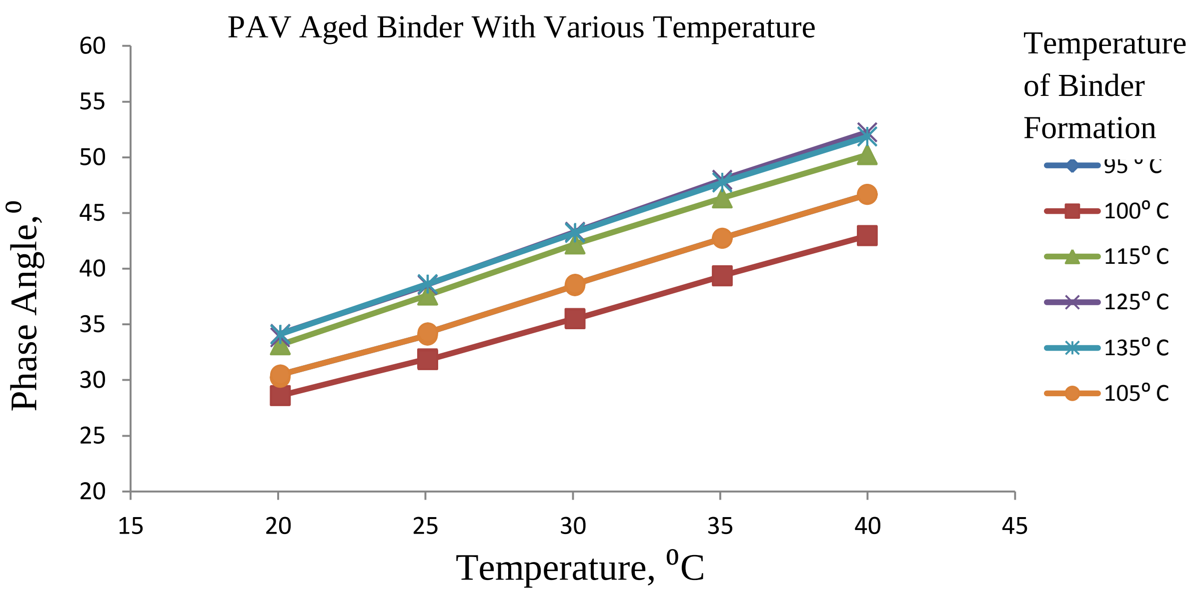

Figure 5.29 Phase Angle versus Temperature

CHAPTER-6

CONCLUSIONS

A number of additives have been tried to modify the engineering properties of bitumen and use the modified binders in paving mixes to derive the maximum benefits to with stand the heavy stresses due to the wheel loads of the modern day traffic. Sulphur is one additive which is found to extend the properties of the binder and this improves the engineering properties of paving mixes. In this project work, sulphur has been added to hot VG 30 bitumen maintaining a particular temperature and manual stirring for about 10 minutes is resorted to result a homogeneous product. To ascertain the optimum quality of the binder, the temperature of mixing was varied from 95° C to 135° C and the sulphur concentration from 0% to 8%. Various rheological properties have been studied for binders thus prepared under both aged and unaged conditions. From the test results obtained and analysis of the work done.

The following concluding remarks have been drawn:

- Considering the complex modulus and other parameters, addition of 2% sulphur by weight of VG 30 bitumen blended at 100° C temperatures results in optimum.
- The additions of sulphur to the extent of 2% conventional bitumen improve the viscoelastic behaviour of the binder.
- The modified binder is also observed to offer superior visco elastic and other rheological characteristics in case of aged binders.

Future scope of the research

- Use scanning electron microscope images to evaluate binder aggregate adhesion.
- Use time sweep test with different model to determine the fatigue life.
- Calculate fatigue life with dissipated energy method.
- Compare the rheological data's through three mechanical tests, indirect tensile strength, indirect tensile resilient modulus, fatigue test.

REFERENCES

1. AASHTO Provisional Standards: AASHTO T315-08, (2011), “Standard Test Method for Determining the Rheological Properties of Asphalt Binder Using a Dynamic Shear Rheometer (DSR)”, Washington.
2. ASTM D5 / D5M-13, “the standard test method for penetration of bituminous materials”.
3. ASTM D113 – 07, “the standard test method for ductility of bituminous materials”.
4. ASTM D36 / D36M – 12, “the standard test method for softening point of bitumen (ring-and-ball apparatus)”.
5. ASTM D70-03, “the standard test method for specific gravity and density of semi-solid bituminous materials (pycnometer method)”.
6. ASTM Standards D 8, “terminology relating to Materials for Roads and Pavements”.
7. ASTM Standards D 2872, “Test Method for Effect of Heat and Air on Rolling Film of Asphalt (Rolling Thin-Film Oven Test)”.
8. Airey, G.D. (1997), “rheological characteristics of polymer modified and aged binders”, unpublished PhD thesis, University of Nottingham.
9. Al-Ansary, (2010), “innovative solutions for sulphur in Qatar”, presented at the sulphur institutes, sulphur world symposium, Doha, Qatar.
10. Al-mehthel, M. et al (2010), “sulphur extended asphalt as a major outlet that outperformed other asphalt mixes in the Gulf”, presented at sulphur institutes, sulphur world symposium, Doha, Qatar.
11. Bahia, H. and Anderson D.A. (1993), “the new proposed rheological properties of asphalt binders”, ASTM Conference, Physical Properties of Asphalt Cement Binders, Texas, pp.1-27.

12. Bahia, H. and Davies, R. (1994), "Effect of crumb-rubber modifiers on performance-related properties of asphalt binders", proceedings of the Association of Asphalt Paving Technologists, Volume 63, pp.414-449.

13. Bahia, H. A. (1995), "Critical Evaluation of asphalt modification using strategic highway research program concepts", Transportation Research Record Vol.1488, Washington D.C., pp. 82- 88.

14. Beaudoin, J.J. and Sereda, P.J. (1979), "a two continuous-phase sulphur-asphalt composite-development and characterisation", Canadian journal of civil engineering, vol.6, no.3, pp 406-412.

15. Fritchy, G. et al (1980), "sulphur as supplementary filler for bitumen- silica composites", rheological acta, vol-20.

16. Fritchy, G. (1980), "sulphur modified bitumen: a new binder", rheological acta, vol-20.

17. Goodrich, J.L (1988), "Asphalt and polymer modified asphalt properties related to the performance of asphalt concrete mixes", Journal of the Association of Asphalt Pavement Technologists, vol 57, pp.116-160.

18. Garcia-Morales. et al (2005), "Effect of waste polymer addition on the rheology of modified bitumen".

19. Hayner, et al (1999), "sulphur treated asphalt compositions and their preparation", patent no. - 6001162.

20. David, R. and Thomas, W. (1994), "THE ASPHALT MODEL: Results of the SHRP Asphalt Research Program", SHRP A-001 Contract, Centre for Transportation Research, University of Texas, Austin, USA.

21. Kumar, P. et al (2009), " rheological properties of crumb rubber modified bitumen- a lab study", journal of scientific and industrial research, vol-68, pp 812-816.
22. Mahrez, A. and Mohamed, R. (2003), "Rheological Evaluation Of Ageing Properties Of Rubber Crumb Modified Bitumen", Journal of the Eastern Asia Society for Transportation Studies, Vol.5.
23. Mohanty, M. (2012), "performance based characterisation of bituminous binder", unpublished b-tech thesis, national institute of technology, Rourkela.
24. Richard, (1965), "the rheology of asphalt, unpublished master of science thesis", Massachusetts institute of technology.
25. Strickland, et al A Study of the Low-Temperature Properties of Sulphur Extended Asphalt Mixtures
26. Strickland, D. et al (2007) "Performance Properties of Sulphur Extended Asphalt Mixtures with SEAM" Shell Bitumen.
27. Singh, M. (2012), "laboratory study on bitumen modification for improved physical, rheological properties and the effect of short term aging", 25th ARRB conference, Australia.
28. Vaidya, et al (2010), A Study On Performance Graded Sulphur Modified Asphalt Compositions For Super Pave, Publication Number:- US 2012/0108708.
29. Van der Poel, C. (1954), "A general System Describing the Viscoelastic Properties of Bitumen and its Relation to Routine Test", Journal of Applied Chemistry, vol 4, pp.221-232.
30. Whiteoak, C. D. (1990), The Shell Bitumen Handbook.